



**Statement of Work
For Analytical Measurements**

WATER QUALITY PARAMETERS

MODULE SS06-B.3

March 9, 1999

Approved: _____
Analytical Services

Reviewed For Classification

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WATER QUALITY PARAMETERS MODULE

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WATER QUALITY PARAMETERS MODULE

INTRODUCTION

This module provides the technical requirements, analytical chemistry services, quality control procedures, and an analysis structure that will generate data of known and documented quality for the identification and quantitation of water quality parameters. The samples to be analyzed may contain potentially hazardous radioactive, inorganic, and/or organic materials. Sample matrices may consist of groundwater, surface water, waste waters, watery sludges, and other aqueous liquids as appropriate for the methods.

Procedures specified herein shall be used in the preparation and analysis of samples for the presence and quantitation of Water Quality Parameters. The Laboratory shall employ safe handling procedures, obtain the required licensing for handling such waste, utilize generally accepted good laboratory practices in the performance of contract requirements, and shall follow the quality assurance/quality control (QA/QC) program specified herein and as required by the General Laboratory Requirements Module, GR01.

In general, method requirements for Water Quality Parameters for the Site are consistent with those specified in the USEPA Test Methods for Evaluating Solid Waste (SW-846), USEPA Methods for the Chemical Analysis of Water and Wastes (EPA-600), U.S. Environmental Protection Agency (USEPA) Contract Laboratory Program SOWs (CLP-SOW), and other nationally-accepted analytical methods or protocols. Updates to these methods shall be used by the Laboratory once the methods are promulgated and approved for use by the Site. The CTR shall provide the Laboratory with implementation dates for any new methods.

The following modules are required for the analysis of Water Quality Parameters under this subcontract: the General Laboratory Requirement Module, GR01; the Requirements for Analytical Services Electronic Deliverable Module, GR02; and the Requirements for Water Quality Parameters Module, SS06. The specifications in SS06 shall supersede any GR01 specifications in the case of conflicting requirements.

EXHIBIT A
SUMMARY OF REQUIREMENTS

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WATER QUALITY PARAMETERS

SUMMARY OF REQUIREMENTS

1. GENERAL REQUIREMENTS

This module comprises the eight exhibits and two appendices which define requirements for the determination of Water Quality Parameters (WQPs). Exhibit A provides an overview of the Water Quality Parameters module and its general requirements. Exhibit B contains reporting and deliverables requirements. Exhibit C contains the Target Analyte List (TAL) and Required Detection Limits (RDL) for all target analytes. Exhibit D specifies analytical procedures, defines the application of these procedures, and contains method specific QA/QC requirements. Exhibit E contains general and specific laboratory QA/QC requirements. Exhibit F contains the evidentiary requirements including, chain-of-custody and evidentiary document control requirements that the Laboratory must follow in processing samples under this subcontract, and specifies requirements for written laboratory Standard Operating Procedures (SOPs). To ensure proper understanding of language utilized in this subcontract, Exhibit G contains a glossary of terms which supplement the glossary found in GR01 Exhibit G. When a term is used in the text without definition, the glossary meaning shall be applicable. Exhibit H contains references applicable to SS06.

2. FACILITY, INSTRUMENTATION AND KEY POSITION REQUIREMENTS

- 2.1. **Facility:** The laboratory facility shall meet all requirements of base analytical methods and the Laboratory Health and Safety Program specified in GR01.
- 2.2. **Instrumentation:** The Laboratory shall have sufficient analytical equipment and capability to meet all terms and conditions of this module and GR01, including all equipment requirements specified in base methods used to perform the analyses. At a minimum, the Laboratory shall have all of the following equipment and instruments operative at the time of the on-site evaluation and committed for the full duration of the subcontract:
 - 2.2.1. UV-Visible Spectrophotometer and/or Chemical Flow Analyzer
 - 2.2.2. Four-place balance
 - 2.2.3. Oven for drying solids at both 103°C and 180°C, and furnace for drying solids residue at 550°C
 - 2.2.4. Exhaust hood with hot plates
 - 2.2.5. Incubator
 - 2.2.6. Refrigerator
 - 2.2.7. ASTM Type II water supply
 - 2.2.8. Meters for pH, conductivity, dissolved oxygen (DO), Ion Selective Electrode (ISE), and turbidity
 - 2.2.9. Autotitrator, or appropriate titration glassware

2.2.10. Ion Chromatograph equipped to analyze target WQP and software to identify and quantify analyte peaks

2.2.11. Infrared Spectrometer and Organic Carbon Analyzer

- 2.3. **Key Position Requirements:** The Laboratory shall designate and use technical key positions listed below and in GR01 to perform the minimum functional requirements necessary to meet the terms and conditions of this subcontract. Minimum academic training and experience qualifications for positions specific to SS06 are identified below. All positions listed in GR01 Exhibit A Section 1, the Sample Custodian and Document Control Officer specified in GR01 Exhibit F Section 4, and all of the positions listed below are considered key positions for this subcontract.

2.3.1. Ion Chromatograph (IC) Chemist Qualifications

Responsibility:	Responsible for analytical procedures, maintenance of IC instrumentation and interpretation of IC data.
Academic Training:	A minimum of a bachelor's degree in a science discipline. Experience may not be substituted for this educational requirement.
Experience:	A minimum of two years of experience with IC analysis of environmental samples.

2.3.2. IC Operator Qualifications

Responsibility:	Responsible for operation and maintenance of IC instrumentation.
Academic Training:	A minimum of a bachelor's degree in a science discipline.
Experience:	A minimum of one year of experience in operating and maintaining IC instrumentation.

2.3.3. WQP Classical Techniques Chemist Qualifications

Responsibility:	Responsible for WQP analysis procedures; maintenance of classical techniques instrumentation and equipment; and review and approval of data.
Academic Training:	A minimum of a bachelor's degree in a science discipline.
Experience:	A minimum of three years of experience in performing colorimetric, gravimetric, potentiometric, titrimetric, and turbidimetric analyses; and maintaining the instrumentation and equipment for these analyses.

2.3.4. WQP Classical Techniques Analyst Qualifications

Responsibility:	Responsible for preparation and analysis of environmental samples.
Academic Training:	A minimum of a high school diploma and a college-level course in general chemistry.
Experience:	A minimum of one year of experience in an analytical laboratory with experience in sample preparation and analysis with WQP analysis methods.

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

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REPORTING AND DELIVERABLES REQUIREMENTS

1. INTRODUCTION

SS06 Exhibit B contains reporting and deliverables requirements applicable to all Water Quality Parameters line item codes defined in SS06 Exhibit C. This exhibit supplements and augments the fundamental deliverable requirements specified in GR01 Exhibit B. Requirements for Sample Data Packages, Supporting Data Packages, and other deliverables specific to Water Quality Parameters are detailed. Three tables in SS06 Exhibit B further define some of the deliverables specified in GR01 Exhibit B:

Table B1. Sample Data Package Deliverables

Table B2. Other Deliverables

Table B3. Supporting Documentation Package Deliverables

These tables define major deliverable components as *Deliverable Sections* which are each assigned titles. Text accompanying the tables provides structural and content requirements. Tables and text also provide reference lists for Water Quality Parameters deliverable requirements found in this module and other modules of this SOW.

SS06 Exhibit B also contains requirements for Document Inventories for Water Quality Parameters.

Deliverable requirements and schedules specified in the General Laboratory Requirements Module, GR01, and its tables shall be met. However, where requirements and schedules contained in SS06 are in conflict with Module GR01, requirements in SS06 shall take precedence.

2. SAMPLE DATA PACKAGE REQUIREMENTS

- 2.1. **Sample Data Package Components:** Table B1 lists the required deliverable sections for a Sample Data Package for Water Quality Parameters. Each deliverable section is numbered and assigned a title. These *Deliverable Section Numbers* and *Titles* are referenced in the remainder of the accompanying text of this SS06 Exhibit B Section 2. The *Reference* column in Table B1 contains designators which refer to modules, exhibits, and sections where more details may be found. This reference column is intended as an aid in locating requirements, but is not expected to be all-inclusive.

TABLE B1 SAMPLE DATA PACKAGE DELIVERABLES

Deliverable Section Number	Deliverable Section Title	Reference (Module, Exhibit/Section Letter or Title)
1	Sample Data Package Cover Page	GR01, Exhibit B/Section 4
2	Table of Contents	GR01, Exhibit B/Section 4
3	Data Review Checklist	GR01, Exhibit B/Section 4 SS06, Appendix A SS06, Exhibit B/Section 2
4	COC(s)	GR01, Exhibit B/Section 4 GR01, Exhibit F/Section 2

TABLE B1 SAMPLE DATA PACKAGE DELIVERABLES (continued)

Deliverable Section Number	Deliverable Section Title	Reference (Module, Exhibit/Section Letter or Title)
5	Narrative	GR01, Exhibit B/Section 4 SS06, Exhibit B/Section 2 SS06, Appendix A/Section 5
6	Sample and QC Result Summaries	GR01, Exhibit B/Section 4, 7, 8 SS06, Exhibit B/Section 2 SS06, Exhibit C SS06, Exhibit E/Section 4 SS06, Appendix A/B
7	Preparation Raw Data	GR01, Exhibit B/Section 4 GR01, Exhibit E/Section 5 GR01, Exhibit F/Section 4 SS06, Exhibit B/Section 2 SS06, Exhibit F/Section 4
8	Standards Summary	GR01, Exhibit B/Section 4 GR01, Exhibit E/Section 6 SS06, Exhibit E/Section 4
9	Instrument Raw Data	GR01, Exhibit B/Section 4 GR01, Exhibit F/Section 4 SS06, Exhibit B/Section 2 SS06, Exhibit F/Section 4
10	Electronic Data Deliverable (EDD) - Hard Copy	GR01, Exhibit B/Section 4 GR02

2.2. Sample Data Package General Requirements

- 2.2.1. All Sample Deliverable Sections shall meet the general and specific requirements listed in GR01 Exhibit B Section 4.
- 2.2.2. All Sample Deliverable Sections shall appear in the Sample Data Package in numerical order by *Deliverable Section Number*.
- 2.2.3. *Deliverable Section Numbers* 6 through 9 shall each be preceded by a Cover Sheet which is titled exactly as given under the *Deliverable Section Title* column of Table B1. These Cover Sheets shall be paginated along with the rest of the Sample Data Package. Each of these Deliverable Section Cover Sheets shall comply with the following structural requirements:
 - 2.2.3.1. The Deliverable Section Title shall appear at the top
 - 2.2.3.2. If the locations of required items within the Sample Data Package differ from the specified location, then these discrepancies shall be mapped in table form on the deliverable section Cover Sheet for the specified location. This table shall include mapping information in columns labeled "Required Item," "Specified Location," and "Actual Location."
 - 2.2.3.3. If identifiers for required items differ from specified identifiers, then these discrepancies shall also be mapped in table form on the Cover Sheet of the affected deliverable section. This table shall include mapping information in columns labeled "Specified Identifier" and "Identifier Used."

- 2.2.4. Any undocumented misplaced items shall be considered incomplete.
- 2.2.5. All raw data from failed analytical batches or from failed individual analyses shall be clearly labeled as "Data Not Used" and shall be included in the appropriate Deliverable Section.
- 2.2.6. Refer to GR01 Exhibit B and SS06 Exhibits D and E for additional requirements for Sample Data Packages.
- 2.3. **Sample Data Package Cover Page Requirements**
(Requirements for Sample Data Package Deliverable Section Number 1)
Sample Data Package Cover Pages shall be included as specified in GR01.
- 2.4. **Table of Contents**
(Requirements for Sample Data Package Deliverable Section Number 2)
A Table of Contents for the Sample Data Package shall be included as specified in GR01.
- 2.5. **Data Review Checklist Requirements**
(Requirements for Sample Data Package Deliverable Section Number 3)
Data Review Checklists document the completeness and the quality control status of the Sample Data Package. SS06 Appendix A contains the form which must be used to complete this check for Water Quality Parameters analyses. A completed Data Review Checklist shall be submitted with each Sample Data Package and shall be in strict conformance with the formatting and content of the form contained in SS06 Appendix A. Refer to General Laboratory Requirements Module GR01 Exhibit B Section 4 for more details.
- 2.6. **Chain of Custody (COC)**
(Requirements for Sample Data Package Deliverable Section Number 4)
COC documentation shall be included in the Sample Data Package as specified in GR01.
- 2.7. **Sample Data Package Narrative**
(Requirements for Sample Data Package Deliverable Section Number 5)
 - 2.7.1. Sample Data Package Narratives shall be included in the Sample Data Package as specified in GR01.
 - 2.7.2. The narrative must contain a table that lists the approved method used for each parameter analyzed. Alternately, method numbers may be included in an additional column on WQP Form 1. In this case, the narrative shall state that *method identifiers are included on WQP Form 1*.
 - 2.7.3. Additional content requirements are listed in GR01 Exhibit B, SS06 Exhibit D, SS06 Appendix A and in other SS06 exhibits.
- 2.8. **Sample And QC Sample Results Summary**
(Requirements for Sample Data Package Deliverable Section Number 6)
 - 2.8.1. The sample and QC sample results summary shall include forms providing observed results and information required to verify calculations for Water Quality Parameters QC analyses. SS06 Exhibit D contains many clarifications and protocols for data which are reported in this section of the deliverable.

- 2.8.2. Sample Data Packages are complete reports analogous to CLP inorganics data packages. Formats and instructions for reporting water quality parameters are specified in SS06 Appendix B. Forms 1 through 7 from SS06 Appendix B shall be included in each Sample Data Package.
- 2.8.3. The Laboratory shall ensure that all forms submitted as equivalent shall include all of the same information present on the Appendix B Forms.
- 2.8.4. At a minimum, forms shall be included which provide summaries for all of the following items:
- Sample results
 - Initial and continuing calibration verification (ICV, CCV) results and recoveries
 - Preparation blanks
 - Initial and continuing calibration blank (ICB, CCB) results
 - Spike sample results and recoveries
 - Duplicate sample results
 - Laboratory Control Sample results and recoveries
 - Sample Holding Time Summaries

2.9. **Preparation Raw Data Deliverable Requirements**

(Requirements for Sample Data Package Deliverable Section Number 7)

- 2.9.1. Sample preparation raw data shall be documented in the form of preparation bench sheets and/or preparation logs. These documents shall follow the requirements defined in SS06 Exhibit F Section 4 and the General Laboratory Requirements Module GR01 Exhibit F Section 4.
- 2.9.2. Preparation Logs shall be included in the deliverable in order by parameter as listed in SS06 Exhibit C Table C1.
- 2.9.3. These logs must include the following:
- Analytical Batch identifier (see the definition of Analytical Batch in GR01 Exhibit G)
 - Date and time of preparation
 - Identifier for the laboratory SOP for the preparation performed
 - Identifiers for all samples, sample QC, and QC solutions prepared
 - Balance identifiers with dates of use or balance verification bench sheet so that all measurements can be traced to the documented balance verifications performed according to GR01 Appendix E Section 5.
 - Initial and final weights and volumes for all samples and QC samples including gross weights and tare weights where applicable
 - Pipette identifiers and dates of use (if applicable)
 - Comments describing any significant sample changes or reactions which occur during preparation and storage
 - Signatures and dates of all analysts and reviewers

2.10. **Standards Summary**

(Requirements for Sample Data Package Deliverable Section Number 8)

Each Sample Data Package shall include documentation which lists identification numbers and expiration dates for all standards used for data reported in the RIN. This documentation shall include secondary standard identification numbers for all secondary standards used for calibration, calibration verifications, matrix spikes and laboratory control solutions. Primary standards used as received shall be identified by their unique identifier. The standards summary documentation included in the Sample Data Package shall also include identifiers for all primary standards used to make secondary standards. These identifiers must be traceable to standard and reference materials certificates included in the Supporting Documentation Package (see SS06 Exhibit B Section 5).

2.11. **Instrument Raw Data**

(Requirements for Sample Data Package Deliverable Section Number 9)

For each reported value, the Laboratory shall include all raw data from each instrument used to obtain reported values for all samples and QC solutions. These raw data shall include instrument readouts and run logs. Raw data shall contain all instrument readouts used for the sample results, including those readings that may fall below the RDL. All instruments must provide a legible hard copy of the direct real-time instrument readout. Numerical readouts shall be printed so that actual calculated or raw values are printed for those readings falling below zero. Originals of the chronological instrument readout must be included. (Copies of original documents may be substituted if the original data were previously submitted with another RIN.)

2.11.1. Raw Data shall be included in order by parameter as listed in SS06 Exhibit C Table C1.

2.11.2. Raw data shall meet labeling and other requirements defined in SS06 Exhibit F Section 4 and the GR01 Exhibit F Section 4. All of the following items must be included in the instrument raw data or in run logs.

- Calibration data, including instrument readouts, slopes, intercepts, correlation coefficients
- All sample data
- Initial and continuing calibration blanks
- Initial and continuing calibration verification standards
- Laboratory spikes, duplicates, and laboratory control samples
- Retention times for ion chromatography
- Autosampler positions, if applicable
- Instrument identifier, instrument adjustments, data corrections or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation.
- Time and date of each analysis. If the instrument output cannot be set to automatically provide the time of analysis, the dates and times of instrument calibration/standardization and final calibration verification must be recorded.
- Dilutions and volumes applied during the instrument analysis.

2.12. **Electronic Data Deliverable (EDD) - Hard Copy**

(Requirements for Sample Data Package Deliverable Section Number 10)

The EDD Hard Copy shall be included as specified in GR01, Exhibit B, Section 4.

3. **QUICK TURN PACKET REQUIREMENTS**

Quick-turn Packets designated in priority processing and rush processing schedules in GR01 shall include the following items from Table B1. Sample Data Package Deliverables:

- Section 4, COC
- Section 5, Narrative
- Section 6, Sample and QC Result Summary, Water Quality Parameters Reporting Form I only for each sample

Priority and Rush processing requires the Quick-turn Packet delivered according to the schedule in GR01 Exhibit B Section 2. Please note that submittal of the Quick-turn Packet does not affect deliverable requirements for Sample Data Packages, QC Result Summaries, Electronic Data Deliverables, Supporting Documentation Packages, or any other deliverables.

4. REQUIREMENTS FOR OTHER DELIVERABLES

The following table defines other required deliverables in addition to the Sample Data Package deliverables for Water Quality Parameters.

TABLE B2 OTHER DELIVERABLES

Deliverable Title	Schedule	Recipient	Reference (Module, Exhibit/Section)
Performance Evaluation Sample Analysis Results	7 days from receipt of results. Minimum frequency quarterly.	CTR	GR01, Exhibit B/Sections 2, 9 GR01, Exhibit E/Section 9 SS06 Exhibit E/Section 7
Electronic Data Deliverable (EDD)	With each Sample Data Package	CTR	GR02 SS06, Exhibit B/Section 4

- 4.1. **Electronic Data Deliverable (EDD):** Requirements for the Electronic Data Deliverable (EDD) are specified in Module, GR02. An EDD is required for all analyses performed under this SOW module, unless otherwise directed in writing by the CTR.

5. SUPPORTING DOCUMENTATION PACKAGE REQUIREMENTS

- 5.1. **Support Package Schedules and Maintenance:** See GR01 Exhibit B Section 5 for delivery schedules and maintenance requirements for Supporting Documentation Packages (Support Packages).
- 5.2. **Support Package Components:** Table B3 lists required components for a Support Package for Water Quality Parameters. Each section is assigned a title. These titles are referenced in the remainder of the accompanying text in SS06 Exhibit B Section 5. The *Reference* column in Table B3 refers to the module, exhibit, and section number where more details may be found. The information in this column is intended as an aid in locating specifics, but may not be all-inclusive. Refer to Exhibits B, E, and F of the General Laboratory Requirements Module GR01 for details about these requirements.

TABLE B3 SUPPORTING DOCUMENTATION PACKAGE

Deliverable Section Title	Reference (Module, Exhibit/Section Number)
Document Inventory	SS06, Exhibit B/Section 5
Sample receipt, storage, tracking and internal COC records	GR01, Exhibit F/Sections 2, 3 SS06, Exhibit D/Section 3 SS06, Exhibit F/Section 3
Copy of Sample Data Package	GR01, Exhibit B/Sections 3, 4, 5 SS06, Exhibit B/Sections 3, 4
Original SRM Certificates	GR01, Exhibit E/Section 6 SS06, Exhibit E/Section 4
Original logs and/or logbooks	GR01, Exhibit F/Section 4 SS06, Exhibit F/Section 4
Standard Operating Procedures	GR01, Exhibit F/Section 6 SS06, Exhibit F/Section 5

TABLE B3 SUPPORTING DOCUMENTATION PACKAGE (continued)

Deliverable Section Title	Reference (Module, Exhibit/Section Number)
MDL Determinations	SS06, Exhibit D/Section 11
Software Quality Assurance	GR01, Exhibit F/Section 5 SS06, Exhibit E/Section 6
Electronic Data Deliverable (EDD)	GR02 SS06, Exhibit B/Section 8

5.3. Document Inventory Requirements:

5.3.1. The Document Inventory shall include a list of all documents in the Support Package file and the locations of all documents that are not physically in the Support Package file, but are listed in Table B3.

5.3.2. The Document Inventory shall appear at the beginning of the Support Package file and shall be the first item in the Support Package. All items listed in the Document Inventory shall be titled exactly as given under the "Deliverable Section Title" column of Table B3. All other items listed in Table B3 may follow in any order, however, the other documents shall appear in the order listed on the Laboratory's Document Inventory.

5.3.3. See GR01 Module B Section 5 for additional Document Inventory requirements for Supporting Data Packages.

5.4. Support Package Internal COC Requirements: The Support Package shall include sample receipt, storage, tracking, and internal COC records required in GR01 Exhibit F Sections 2 and 3.

5.5. Support Package Copy of the Sample Data Package: The Support Package shall include a photocopy of the Sample Data Package.

5.6. Support Package Original SRM Certificate Requirements: The Support Package shall include the originals of all standard certificates required for meeting GR01 Exhibit E Section 6 and SS06 Exhibit E Section 3. It is expected these originals will be included by referencing their location on the Document Inventory.

5.7. Support Package Logs and Logbook Requirements: Logs and/or logbooks include, but are not limited to, preparation logs, instrument run logs, standard dilution logs, balance calibration logs, pipette calibration logs, instrument maintenance logs, and ASTM II water logs must all be included in the Support Package. (It is expected that many of these items will be included by referencing their location on the Document Inventory.)

5.8. Support Package Standard Operating Procedures: The Support Package shall include all SOPs required for meeting SS06 Exhibit F Section 5. (It is expected these procedures will be included by referencing their identifiers and location on the Document Inventory.)

5.9. Support Package MDL Determinations: The location of original raw data and a data summary sheet for all MDL determinations relevant to the RIN shall be listed on the Document Inventory for the RIN. (It is expected that these data will be included by referencing their location on the Document Inventory.) These referenced data shall meet all requirements listed in SS06 Exhibit F Section 4 and GR01 Exhibit F Section 4. At a minimum, MDL data summary sheet(s) shall list the following:

- Dates for MDL determinations,
- 'True' values for solutions analyzed for MDL determinations,

- Mean values calculated for each MDL analysis date (in concentration units)
 - Standard deviations calculated for each MDL analysis date (in concentration units)
 - Calculated MDLs
- 5.10. **Support Package Software Quality Assurance:** The Support Package shall include Software Quality Assurance Documentation required by GR01, Exhibit F Section 5. (It is expected that this information will be included by referencing its location on the Document Inventory.)
- 5.11. **Support Package Electronic Data Deliverable Requirements:** The copy of the Sample Data Package contained in the Support Package may include the EDD by reference to the physical location and file identifier for a retained copy of the EDD.

EXHIBIT C
WATER QUALITY PARAMETERS
AND
REQUIREMENTS

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WATER QUALITY PARAMETERS REQUIREMENTS

1. WATER QUALITY PARAMETERS REQUIREMENTS LISTS

Table C1 and Table C2 specify required parameters, required detection limits (RDLs), required method sources and method type descriptions for each WQP line item code.

2. TABLE C1 AND TABLE C2, WATER QUALITY PARAMETERS

Table C1 WATER QUALITY PARAMETERS

Line Item Codes	Parameter Identifier	Parameter Name	RDL ⁽¹⁾ (mg/L)	Approved Methods ⁽²⁾	Method Type
SS06B001	10-70-8	Acidity	10	EPA 305.1, SM 2310	Titrimetric
SS06B002	T-005	Alkalinity, Total as CaCO ₃	10	EPA 310.1, 310.2, SM 2320 B	Titrimetric (to pH 4.5)
SS06B003	71-52-3	Alkalinity, Bicarbonate (HCO ₃ ⁻) as CaCO ₃	10	EPA 310.1, 310.2, SM 2320 B	Titrimetric
SS06B004	3812-32-6	Alkalinity, Carbonate (CO ₃ ²⁻) as CaCO ₃	10	EPA 310.1, 310.2, SM 2320 B	Titrimetric
SS06B005	7727-37-9	Ammonia as N	0.1	EPA 350.1, SM 4500-NH ₃ H	Colorimetric/Spectrophotometric (Automated-Phenate)
				EPA 350.3, 4500-NH ₃ -F, G,	Potentiometric (Ion Selective Electrode)
SS06B006	10-26-4	Biochemical Oxygen Demand (BOD ₅)	2	HACH GRAPHICAL METHOD	Potentiometric (Dissolved Oxygen Depletion)
SS06B007	24959-67-9	Bromide	2	EPA 300.0	Ion Chromatography
				EPA 320.1	Titrimetric
SS06B008	11-03-0	Carbonaceous Biochemical Oxygen Demand (CBOD ₅)	2	HACH GRAPHICAL METHOD	Potentiometric (Dissolved Oxygen Depletion with Nitrification Inhibitor)
SS06B009	C-004	Chemical Oxygen Demand (COD)	5	EPA 410.4, SM 5220 D	Colorimetric/Spectrophotometric
SS06B010	16887-00-6	Chloride	0.5	EPA 300.0	Ion Chromatography
				EPA 325.3	Titrimetric
SS06B011	18540-29-9	Chromium VI (Hexavalent Cr)	0.005	EPA 218.4, SM3500-Cr D, SW-846 7196 A,	Colorimetric/Spectrophotometric
SS06B012	57-12-5	Cyanide, Total	0.005	EPA 335.3, 335.4, SM4500-CN C, E	Colorimetric/Spectrophotometric (Manual Distillation followed by Analysis)
SS06B013	57-12-5	Cyanide, Total, for RCRA Compliance	0.005	SW-846 9010, 9012	Colorimetric/Spectrophotometric (Manual Distillation followed by Analysis)
SS06B014	10-87-7	Cyanide, Amenable to Chlorination	0.005	EPA 335.1, SM4500-CN G	Colorimetric/Spectrophotometric
SS06B015	10-87-7	Cyanide, Amenable to Chlorination, for RCRA Compliance	0.005	SW-846 9010A & 9012	Colorimetric/Spectrophotometric
SS06B016	Inactive	Inactive	Inactive	Inactive	Inactive

Table C1 WATER QUALITY PARAMETERS (continued)

Line Item Codes	Parameter Identifier	Parameter Name	RDL ⁽¹⁾ (mg/L)	Approved Methods ⁽²⁾	Method Type
SS06B017	10-71-9	Cyanide, Releasable, for RCRA Compliance	0.005	SW-846 Chapter 7, SW-846 9010A, & 9012	Colorimetric/Spectrophotometric (Distillation followed by Analysis)
SS06B018	16984-48-8	Fluoride	0.5	EPA 300.0	Ion Chromatography
				EPA 340.2, SM4500-F B, C	Potentiometric (Distillation followed by ISE)
SS06B019	11-02-9	Hardness as CaCO ₃	10	EPA 130.2, SM 2340C	Titrimetric
SS06B020	14797-55-8	Nitrate as N	0.5	EPA 300.0	Ion Chromatography
				EPA 352.1, EPA 353.1, 353.2,	Colorimetric/Spectrophotometric (Brucine sulfate) (NO ₃ /NO ₂ less.2 NO ₂)
SS06B021	14797-65-0	Nitrite as N	0.5	EPA 300.0	Ion Chromatography
				EPA 354.1, SM4500-NO ₂ ⁻ B, EPA 353.1, 353.2, 4500-NO ₃ ⁻ E, H	Colorimetric/Spectrophotometric (Without reduction)
SS06B022	C-005	Nitrate/Nitrite as N (Total Nitrate/Nitrite as N)	0.05	EPA 353.1, SM4500-NO ₃ ⁻ H, EPA 353.2, 4500-NO ₃ ⁻ E	Colorimetric/Spectrophotometric (Auto Hydrazine) (Cadmium Reduction)
SS06B023	10-30-0	Oil and Grease, Total Recoverable	5	EPA 413.1, EPA 413.2	Gravimetric Extraction
SS06B024	11-59-6	Organic Carbon, Dissolved (DOC)	1.0	EPA 415.1	IR
SS06B025	10-35-5	Organic Carbon, Total (TOC)	1.0	EPA 415.1, SM5310 B, C, D	IR
SS06B026	10-29-7	pH (Hydrogen Ion)	0.1 S.U. at 25°C	EPA 150.1, SM4500-H ⁺ B, SW-846 9040 (water), SW-846 9045A (soil)	Potentiometric
SS06B027	108-95-2	Phenol	0.1	EPA 420.1, SM 5530D	Colorimetric/Spectrophotometric
SS06B028	14265-44-2	Phosphate (ortho) as P (Ortho Phosphate)	0.01	EPA 365.1, .2, .3, SM4500-P F, E	Colorimetric/Spectrophotometric
				EPA 300.0	Ion Chromatography
SS06B029	7723-14-0	Phosphate (total)as P	0.01	EPA 365.1, .2, .3 SM4500-P B,5	Colorimetric/Spectrophotometric (Persulfate digestion followed by OrthoPhosphate Analysis)
SS06B030	See Table C-2	Sediment Analysis, Sand-Silt Split	N/A	ASTM D422 & D4822, USGS ⁽³⁾	Gravimetric
SS06B031	7631-86-9	Silica as SiO ₂ , Dissolved	5	EPA 370.1, SM4500-Si D	Colorimetric/Spectrophotometric
SS06B032	11-06-3	Solids, Non-Volatile Suspended (NVSS), (Non-Filterable Residue at 550°C)	5	EPA 160.4, SM2540 E	Gravimetric (TSS-VSS)
SS06B033	C-008	Solids, Total (TS) (Total Residue at 103°C to 105°C)	10	EPA 160.3, SM2540 B	Gravimetric
SS06B034	10-33-3	Solids, Total Dissolved Solids (TDS), (Filterable Residue at 180°C)	10	EPA 160.1, SM2540 C	Gravimetric

Table C1 WATER QUALITY PARAMETERS (continued)

Line Item Codes	Parameter Identifier	Parameter Name	RDL ⁽¹⁾ (mg/L)	Approved Methods ⁽²⁾	Method Type
SS06B035	10-32-2	Solids, Total Suspended (TSS) (Non-Filterable Residue at 103°C to 105°C)	5	EPA 160.2, SM2540 D	Gravimetric
SS06B036	10-34-4	Specific Conductance (Conductivity)	10 mmho/cm at 25°C	EPA 120.1, SM 2510 B	Potentiometric
SS06B037	14808-79-8	Sulfate as SO ₄ ²⁻	5.0	EPA 375.1, EPA 375.2 SW846-9035 & 9036	Colorimetric/Spectrophotometric
				EPA 300.0	Ion Chromatography
SS06B038	RFS-RS-97	Sulfide as H ₂ S, Releasable, for RCRA Compliance	1	SW846 Chapter 7, SW-846 9030A	Titrimetric (Distillation followed by Analysis)
SS06B039	18496-25-8	Sulfide as S	0.002	EPA 376.1, .2 SM4500-S ²⁻ E ⁽⁴⁾	Colorimetric/Spectrophotometric (Gas Dialysis, Automated Methylene Blue Method)
SS06B040	7727-37-9	Total Kjeldahl Nitrogen (TKN) (Organic Nitrogen as N)	0.2	EPA 351.1, 351.2, 351.3, 351.4 SM4500-NH ₃	Colorimetric/Spectrophotometric (Preparation followed by Ammonia as N)
SS06B041	59473-04-0	Total Organic Halides (TOX)	1.0	SW-846 9020,	TOX
SS06B042	10-90-2	Total Petroleum Hydrocarbons (TPH)	1.0	EPA 418.1, SM 5520 F	IR
SS06B043	10-08-02	Turbidity	1.0 NTU	EPA 180.1, SM2130 B	Turbidimetric (Nephelometric)

- (1) RDLs (Required Detection Limits) listed in Table C1 specify maximum allowed levels for MDLs. See SS06 Exhibit D Section 11.
- (2) It is the responsibility of the Laboratory to assure that the method appropriate to the sample matrix and RDL is chosen from the specified method source. The most recently promulgated version should be used unless a specified version requested. The referenced SM methods are from the 18th edition of *Standard Methods for the Examination of Water and Wastewater*.
- (3) 'Laboratory Theory And Methods For Sediment Analysis, Chapter C1 of Techniques of Water-Resources Investigations of the United States Geological Survey,' 1969; Harold P. Guy, U.S. Geological Survey.

'Quality-Assurance Plan For The Analysis Of Fluvial Sediment By Laboratories Of The U.S. Geological Survey,' *U. S. Geological Survey Open-File Report 91-467*, Wilbur J. Matthes, Jr., Clyde J. Sholar, and John R. George, U. S. Geological Survey,
- (4) This referenced SM method is from the 19th edition of *Standard Methods for the Examination of Water and Wastewater*.

Table C2 - Parameter Details for LIC SS06*030

Parameter Identifier	Parameter Name	Parameter Description	RDL
RFS-SS-96-1	Sieve 1	> .75 in	N/A
RFS-SS-96-3	Sieve 3	<.375" & > .188"	N/A
RFS-SS-96-4	Sieve 4	<.188" & > 2mm	N/A
RFS-SS-96-5	Sieve 5	<2mm & >425microns	N/A
RFS-SS-96-6	Sieve 6	<425microns & >75 microns	N/A
RFS-SS-96-7	Remainder	<75 microns	N/A

- Indicates the most current Parameter specific Analytical Module Revision Letter.

EXHIBIT D

ANALYTICAL METHODS

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ANALYTICAL METHODS

1. INTRODUCTION

This Exhibit contains the analytical methods requirements for Water Quality Parameters analysis. The purpose of this section is to provide specific guidance for analyses specified in SS06 Exhibit C. This exhibit is not intended to be a comprehensive requirements document, rather it is intended to clarify and supplement requirements found in approved method sources. In many cases, these instructions apply specific protocols to requirements and recommendations of approved method sources. The objective is to increase uniformity between requirements of different approved method sources while still meeting or exceeding minimum method requirements

2. METHOD SELECTION AND APPLICATION

2.1. **Selection of Base Method:** Methods used for analysis must meet all the following requirements:

- 2.1.1. Methods must be selected from the Approved Methods listed in SS06 Exhibit C Table C1 under the Line Item Code identified for the sample on the COC.
- 2.1.2. Sample pretreatment, digestion, and analysis procedures must be appropriate to the sample matrix and regulatory requirements.
- 2.1.3. The analytical process (including sample pretreatment, digestion, and analysis procedures where applicable) must achieve the RDLs specified in SS06 Exhibit C Table C1 for each listed parameter. These methods must be applied such that the Required Detection Limits (RDLs) in SS06 Exhibit C are achieved. However, if the sample concentration exceeds ten times the method detection limit (MDL) of the instrument or method in use, the value may be reported even though the instrument or method detection limit may not equal the RDL for the specified Line Item Code. (The method employed must be from the Approved Method Source and must comply with all other requirements.)
- 2.1.4. When total cyanide and amenable cyanide for RCRA compliance are requested, the sample must be treated according to SW-846 Method 9010 or 9020 prior to analysis for cyanide.

2.2. **Interpretation of Base Method Requirements:** Analyses performed must meet requirements of the methods selected from the Approved Method Source.

- 2.2.1. The Laboratory must be in strict compliance with all requirements in the these methods which are stated as 'must' or 'shall.'
- 2.2.2. Additional method requirements and quality assurance measures are listed in SS06 Exhibits D and E. In the event method requirements and quality assurance measures in approved methods sources appear to be less stringent than those specified in SS06 Exhibits D or E, the requirements of SS06 Exhibits D and E shall prevail.
- 2.2.3. Gravimetric Solids determinations (e.g., NVSS, TSS, and TDS) by Line Item Codes SS06*032 through SS06*035 shall be performed with enough sample, to a maximum of 500 mL, to yield 1 mg of residue for all Site samples.

2.3. **SW-846 Methods:** For the analysis of samples with Line Item Codes specifying SW-846 as the Approved Methods Source, methods shall be chosen from the most-recently promulgated version of SW-846 as approved by the CTR according to GR01 Exhibit D Section 1. MDLs must be determined as specified in SW-846. Determined MDLs must be less than or equal to the listed

RDL for all Line Item Codes with SW-846 as the Approved Method source. Where samples are diluted in preparation and/or before analysis, MDLs must be multiplied times the combined dilution factors for comparison to RDLs. See SS06 Exhibit D Section 4 for dilution instructions.

- 2.4. **EPA-600 Methods:** For the analysis of samples with Line Item Codes specifying EPA methods other than SW-846 methods, methods shall be chosen from the most-recently promulgated revision of the EPA Methods as approved by the CTR according to GR01 Exhibit D Section 1. Determined MDLs must be less than or equal to the listed RDL for parameters. Where chosen sample preparation methods result in concentration of the samples, RDLs may be multiplied by these concentration factors for comparison to MDLs. For example, if the RDL is listed as one 1 ug/L and the sample preparation results in a two-fold concentration of the sample, an MDL of 2 or less will be acceptable. Where samples are diluted in preparation and/or before analysis, MDLs must be multiplied times the combined dilution factors for comparison to RDLs. See SS06 Exhibit D Section 4 for dilution instructions.
- 2.5. **Method Selection:** It is the responsibility of the Laboratory to choose methods from SS06 Table C1 which are appropriate for the required RDLs and the concentration of the parameter in samples.
- 2.6. **Base Method Modifications:** Standard methods may be modified or alternative methods substituted only with the written consent of the CTR. The requirement of the base methods shall have precedence over SS06 Exhibit D requirements.
- 2.7. **Minimum QC Requirements:** Analyses must be performed with the minimum QC requirements of the base methods and the requirements listed in SS06 Exhibit D. When there is a conflict between SS06 QC requirements, the requirements of the base methods take precedence.

3. SAMPLE HOLDING TIMES AND PRESERVATION REQUIREMENTS

Requirements for sample holding times and preservation of samples for analysis of WQP are specified in 40 CFR §136.3, Table II-*Required Containers, Preservation Techniques, and Holding Times* and in SW-846.

4. GENERAL QUALITY REQUIREMENTS

The following requirements apply to all determinations regardless of the Approved Method Source:

- 4.1. **Analytical Batch Size:** The maximum number of client samples in an analytical batch is twenty for all Water Quality Parameters.
- 4.2. **Measurement Range:** All results shall be taken from measurements made within the calibration curve of the instrument. All analyzed solutions with readings exceeding the highest calibration standard must be diluted and reanalyzed.
- 4.3. **Cleaning protocols:** Cleaning protocols for new and used labware shall meet all requirements specified in base methods.
- 4.4. **Hazardous Additions:** If separation techniques are required for reduction of matrix interference, these techniques shall not introduce hazardous constituents over and beyond those which may be inherent to the sample, when alternative separation methods are available.

- 4.5. **Requirements for Sample Dilutions Due to High Analyte Concentrations:** When sample readings exceed the calibration curve, the sample must be diluted and reanalyzed. Analyte concentrations following dilutions must be greater than the RDL or ten times the MDL, whichever is greater.
- 4.6. **Requirements for Sample Dilutions Performed due to Interference:** Samples containing interferences should be diluted or treated so that the interference effects are diminished as demonstrated by acceptable spike recoveries. Where possible, dilutions shall be chosen so that RDLs are met and interference effects are minimized.
- 4.7. **Standard Traceability:** Standard documentation shall be maintained as stated in GR01 Exhibit B Section 4.
- 4.8. **Labeling Codes:** SS06 Exhibit D Table D2 contains codes to be used for sample and quality control sample labeling.

5. REQUIREMENTS FOR CALIBRATION AND STANDARDIZATION

5.1. Calibration Frequency Requirements

- 5.1.1. Instruments shall be calibrated daily or once every 24 hours and each time the instrument is set up. The Calibration/standardization date and time shall be included in the raw data.
- 5.1.2. For IC analyses, daily calibration is not required, but a calibration curve must be determined whenever one or more of the following conditions exist:
 - 5.1.2.1. The existing calibration curve does not meet calibration verification requirements of SS06 Exhibit D Section 6.
 - 5.1.2.2. The type or concentration of eluent and regenerant is changed.
 - 5.1.2.3. Any instrument parameters such as eluent flow rate are changed.
 - 5.1.2.4. Response or retention times for any analyte varies from those values obtained during calibration by more than ten percent.
- 5.1.3. Concentrations of preparation and preservation reagents in calibration standards and prepared samples must be identical. The laboratory shall introduce calibration solutions as described in the individual methods (see Exhibit C) and record the readings.

5.2. Calibration Curve Requirements For Colorimetric/Spectrophotometric, IC, IR, Potentiometric(ISE), TOX, Turbidimetric Techniques

- 5.2.1. A minimum of four points is required for all calibration curves.
- 5.2.2. Calibration standards shall be prepared at a minimum of three different concentration levels. These three standards and the calibration blank comprise the four-point calibration curve.
- 5.2.3. At least one non-blank standard concentration shall be at the RDL, another standard shall be at a concentration near the mid-point of the calibration curve, and the remaining standard shall define the upper limit of the calibration curve.
- 5.2.4. To establish a calibration curve for each reported target analyte, peak heights, absorbance readings, or area responses are tabulated against the associated standard concentrations.

Using the least squares fit linear regression, the correlation coefficient is calculated for each calibration curve. The correlation coefficient for each reported target analyte must be > 0.995 .

- 5.2.5. These calibration curve requirements must be met even if the calibration curve and concentration calculations must be performed externally to the analytical instrumentation. Documentation of calculations must meet all requirements of SS06 Exhibits B, E, and F.

5.3. **Titrimetric Standardizations**

- 5.3.1. Titrants shall be standardized monthly and the laboratory shall provide the last date of titrant standardization on the bench-sheet. Titrant concentration values shall be traceable to standardization data included in the Supporting Documentation Package (see SS06 Exhibit B, Section 5).
- 5.3.2. For alkalinity by electrometric endpoint, the pH probe shall be calibrated using two pH buffers (four and seven). The calibration shall be documented on the bench sheet and provided as part of the data package deliverable.
- 5.3.3. For all other titrimetric determinations, the laboratory shall calibrate the instrument according to the specifications provided by the instrument manufacturer.

5.4. **pH Standardizations**

- 5.4.1. Calibration of the pH meter and electrode system shall consist of a two-point calibration per the instrument manufactures instructions. The two-point calibration must bracket the sample pH for pH measurement greater than 4 and less than 10. Standard pH buffer temperatures during calibration, and the slope and offset values of the calibration curve shall be recorded on the pH Logsheet. If the absolute value of the difference between the measured and true pH buffer values is greater than 0.1 S.U., the pH meter must be recalibrated.

5.5. **Conductivity Standardizations**

- 5.5.1. For conductivity measurements, standardize the instrument with a 0.01 molar standard potassium chloride solution at 25°C per the instrument manufactures instructions. If a difference between the observed value and the standard value of less than 1% can not be obtained, clean the conductivity cell and recalibrate.

5.6. **Dissolved Oxygen Standardizations**

- 5.6.1. For dissolved oxygen (DO) measurements, calibrate the instrument per the instrument manufacturer's instructions.

6. **REQUIREMENTS FOR CALIBRATION VERIFICATIONS**

6.1. **Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) For Colorimetric/Spectrophotometric, IC, IR, Potentiometric (ISE), Titrimetric, TOX, Turbidimetric Techniques**

- 6.1.1. Immediately after calibration of each system, the accuracy of the initial calibration shall be verified and documented for every parameter by the analysis of the Initial Calibration Verification (ICV) solution(s).

- 6.1.2. A Continuing Calibration Verification (CCV) solution must be analyzed and reported for each parameter, at a minimum frequency of one in every 20 measurements and after the last analytical sample.
 - 6.1.3. The parameter concentrations in the ICV and CCV standards shall be at or near the midpoint of the calibration curve. The same standard may be used for both CCV and ICV. Concentrations of preparation and preservation reagents in CCV and ICV standards and prepared samples must be similar.
 - 6.1.4. A NIST-certified solution (or equivalent) must be used for the ICV and CCV solutions. If certified ICV and CCV solutions are not available from any source, the ICV and CCV shall be prepared using an independent standard at a concentration other than that used for instrument calibration and near the midpoint of the calibration curve. An independent standard is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration.
 - 6.1.5. ICV and CCV measurements must reflect the analysis conditions (duration of analysis, rinses, and other related operations) of all associated analytical samples.
 - 6.1.6. When the ICV percent recovery (%R) is outside the control limits of 85% to 115%, the analysis must be stopped, the problem corrected, and the instrument recalibrated.
 - 6.1.7. If the CCV percent recovery (%R) is outside the control limits of 85% to 115%, the analysis must be stopped, the problem corrected, the instrument recalibrated, and affected samples reanalyzed. (Affected samples are defined as all samples analyzed since the last 'in control' ICV or CCV.)
- 6.2. **Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) For Colorimetric/Spectrophotometric, IC, IR, Titrimetric, Potentiometric (ISE), TOX, Turbidimetric Techniques**
- 6.2.1. The ICB and CCB solutions shall be identical to the calibration blank.
 - 6.2.2. The ICB must be analyzed immediately after every ICV.
 - 6.2.3. The CCB must be analyzed immediately after every CCV.
 - 6.2.4. If the absolute value of the ICB or CCB reading is greater than the RDL, the analysis must be stopped, the problem corrected, the instrument recalibrated, and affected samples reanalyzed. Affected samples are defined as all samples analyzed since the last 'in control' ICB or CCB.
 - 6.2.5. Actual results of all ICB and CCB analyses associated with Site samples shall be reported and documented on QC summary forms.
- 6.3. **ICV & CCV For pH:** Calibration verification of the pH meter and electrode system shall consist of the analysis of a standard pH buffer near the mid-point of the calibration line. The temperature of the standard buffer at the time of analysis shall be recorded on the pH logsheet or benchsheet. If the absolute value of the difference between the measured and true pH buffer values is greater 0.10 S.U., the calibration must be repeated and affected samples reanalyzed. (Affected samples are defined as all samples analyzed since the last 'in control' ICV or CCV.)
- 6.4. **ICV & CCV For Conductivity:** A Calibration verification shall be performed with at least one standard potassium chloride solution other than the 0.01M KCl standard solution used for

standardization. When possible, two standard KCl solutions should be analyzed that bracket the sample conductivity. If percent recovery (%R) is outside the control limits of 95% to 105%, for either standard, the instrument must be recalibrated and affected samples reanalyzed. (Affected samples are defined as all samples analyzed since the last 'in control' ICV or CCV.)

- 6.5. **Gravimetric Verifications:** For gravimetric determinations, the laboratory shall provide the analytical balance standardization verification results for the balance check weight measurements prior to (ICV) and following (CCV) analytical batch measurements. The standardization verifications shall meet all requirements of GR01 Exhibit E Section 5.
- 6.6. **Retention Time Windows For Ion Chromatography:** If response or retention times for any IC analyte varies from calibration standards by more than 10%, recalibrate and reanalyze all affected samples. The width of the retention time window used to make identifications should be based upon measurements of the actual retention time variations of standards over the course of the analysis batch. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms. If a resulting chromatogram fails to produce adequate resolution, or identification of specific anions is questionable, confirmatory techniques such as sample dilution and spike must be used.

7. REQUIREMENTS FOR PREPARATION BLANKS

Preparation blanks are required for all methods requiring sample preparation before analysis.

7.1. Preparation Blank Analyses

- 7.1.1. A minimum of one preparation blank must be prepared with each analytical batch requiring sample preparation before analysis. Dilution of samples only for analysis is not to be considered sample preparation. The preparation blank is an aliquot of reagent water or ASTM Type II water treated exactly as a sample including exposure to all labware, equipment, and reagents.
- 7.1.2. Preparation Blank subtraction is not permissible under any circumstance. A target analyte present in both a blank and sample analysis shall be reported in each.
- 7.1.3. Actual results of all blank analyses associated with Site samples shall be reported on QC summary forms.
- 7.1.4. If the PB concentration is greater than the RDL, all associated samples with analyte concentrations less than five times the blank concentration shall be redigested and reanalyzed for that analyte as part of a new complete analytical batch.
- 7.1.5. A BOD/CBOD dilution water-seed blank must be prepared with each BOD/CBOD analytical batch. The blank result (DO uptake) is determined from the difference of the initial DO and the final DO of a BOD bottle full of dilution water and seed aliquot incubated at 5°C for 5 days. The BOD/CBOD dilution water-seed blank uptake shall be reported. No control limit is applicable to the blank uptake for the HACH Method since the HACH Graphical calculation method automatically compensates for the DO uptake of the dilution water-seed blank.

8. REQUIREMENTS FOR MATRIX SPIKED SAMPLES

8.1. **Matrix Spike Sample Frequency For Colorimetric/Spectrophotometric, IC, IR, Potentiometric(ISE), Titrimetric, TOX, Turbidimetric Techniques:** The frequency of preparation and analysis matrix spikes must meet all requirements of base methods. At a minimum, one matrix spike is required in each analytical batch (maximum batch size is twenty samples). Matrix spikes are required for each matrix or waste type within an analytical batch.

8.2. Spike Sample Analysis

- 8.2.1. Sample spikes must be added before sample treatment or preparation begins when sample preparation is required before analysis. Dilution of samples only for analysis is not to be considered sample preparation. A spiked sample must be subjected to the same sample preparation, analytical methods and QA/QC procedures employed for the Site samples. Regardless of whether sample preparation is required, a spiked sample is to be analyzed with each analytical batch.
- 8.2.2. Client samples identified as blanks cannot be used for spiked sample analysis. The Site may require that a specific sample be used for the spike sample analysis.
- 8.2.3. The concentration level of the spike must be equivalent to the mid-point concentration of the instrument calibration curve. For methods not requiring preparation before analysis, spikes shall be added to samples after necessary dilutions have been performed so that spiked and unspiked sample dilution levels are identical.
- 8.2.4. Documentation and reporting of spike sources and concentrations shall meet all requirements of GR01 Exhibit E Section 6.
- 8.2.5. Preparation and Analysis Spike Recoveries:
 - 8.2.5.1. If the spike recovery is within 75% to 125%, when calculated based on original sample and spike results, no additional analyses are required.
 - 8.2.5.2. If the sample analyte concentration is greater than four times the spike amount, quantifiable spike recoveries are not expected; results are acceptable.
 - 8.2.5.3. If the Preparation or Analysis Spike recovery is not within 75% to 125%, when calculated based on original sample and spike results, dilute all samples associated with the spike sample matrix if dilution will not result in analyte-spike measurements being below the method detection limit and reanalyze.
 - 8.2.5.4. If Preparation Spike recoveries are not within 75% to 125%, a post preparation or analysis spike should be completed to determine if the recovery problem is a matrix problem or preparation problem. Whatever the case, the analytical batch shall be reprepared appropriately and reanalyzed. If an acceptable Preparation Spike recovery can not be obtained, report results as directed in SS06 Appendix B for all samples associated with the spike sample matrix.
 - 8.2.5.5. If an Analysis Spike recovery is not within 75% to 125% after sample dilution and/or treatment, when possible, report results as directed in SS06 Appendix B for all samples associated with the spike sample matrix.

9. REQUIREMENTS FOR LABORATORY DUPLICATES

- 9.1. **Laboratory Duplicate Sample Frequency:** The frequency of preparation and analysis of duplicate samples must meet all requirements of base methods. At a minimum, one laboratory duplicate is required in each analytical batch (maximum batch size is twenty samples). Laboratory duplicates are required for each matrix or waste type within a batch. A duplicate sample is to be analyzed with each analytical batch regardless of whether sample preparation is required.
- 9.2. **Duplicate Sample Analysis**
- 9.2.1. Laboratory duplicates must be designated before sample treatment or preparation begins. The laboratory duplicate must be subjected to the same sample preparation, analytical methods and QA/QC procedures employed for the Site samples. Regardless of whether sample preparation is required, a duplicate sample is to be analyzed with each analytical batch.
- 9.2.2. Client samples identified as blanks cannot be used for duplicate sample analysis. The Site may require that a specific sample be used for the duplicate sample analysis.
- 9.3. **Laboratory Duplicate Agreement For Colorimetric/Spectrophotometric, IC, IR, BOD/CBOD, Titrimetric, Potentiometric(ISE), TOX, Gravimetric, Turbidimetric Techniques**
- 9.3.1. The results of the sample and duplicate of the sample must be within a Relative Percent Difference (RPD) of 20% for sample concentrations greater than five times the analyte RDL or the absolute value of the difference between the sample and duplicate results must be less than the RDL for sample concentrations less than five times the RDL.
- 9.3.2. If the RPD of a sample and duplicate involving sample preparation is not within the above control limits, the analyses must be terminated, the problem corrected, and the samples associated with the Laboratory Duplicate reanalyzed (after preparation of another analytical batch, if necessary).
- 9.3.3. If the deviation of a sample and duplicate not involving sample preparation is not within the above control limits, the analyses must be terminated, the problem corrected, and the analytical batch reanalyzed.
- 9.4. **Laboratory Duplicate Agreement For pH:** If the absolute value of the difference between the sample and duplicate sample pH values is greater than 0.10 S.U., the problem must be corrected and the analysis of all affected samples repeated.
- 9.5. **Laboratory Duplicate Agreement Conductivity:** If the RPD between sample and duplicate sample conductivity values is greater than 5% the problem must be corrected and the analysis of all affected samples repeated. (RPD is calculated as directed in SS06 Appendix B WQP Form 5 instructions.)

10. REQUIREMENTS FOR LABORATORY CONTROL SAMPLES

10.1. Laboratory Control Sample Analysis For All Techniques

10.1.1. A Laboratory Control Sample (LCS) must be analyzed for each analyte using the same sample preparation, analytical methods and QA/QC procedures employed for Site samples. Regardless of whether sample preparation is required, an LCS is to be analyzed with each analytical batch for each analytical method and parameter.

10.1.2. The laboratory shall obtain standards traceable to NIST (or equivalent). If such certified standards are not available, the LCS may be prepared using stock certified standards (salts) in ASTM Type II water, or standard grade soil at a different concentration and from a source independent from the ICV/CCV standard.

10.1.3. Documentation and reporting of LCS sources and concentrations shall meet all requirements of GR01 Exhibit E Section 6.

10.2. Laboratory Control Sample Recovery For Colorimetric/Spectrophotometric, IC, IR, Potentiometric (ISE), Titrimetric, TOX, Gravimetric, Turbidimetric Techniques

10.2.1. The LCS concentration shall be within the limits of the calibration curve. For an analytical batch with results determined with autodilution, an LCS also requiring dilution shall be analyzed similar to the samples in the analytical batch.

10.2.2. If the %R of an LCS involving sample preparation is not within the control limits of 80% to 120%, the analyses must be terminated, the problem corrected, and samples associated with that LCS reprepared and reanalyzed.

10.2.3. If the %R of an LCS not involving sample preparation is not within the control limits of 80% to 120%, the analyses must be terminated, the problem corrected, and the analytical batch reanalyzed.

10.3. **Laboratory Control Sample Recovery For pH:** If the absolute value of the difference between the found and true LCS values is greater than 0.10 S.U., the calibration must be repeated. No data associated with the out-of tolerance LCS may be reported.

10.4. **Laboratory Control Sample Recovery For Conductivity:** If the %R of an LCS is not within the control limits of 95% to 105%, the instrument must be recalibrated. . No data associated with the out-of tolerance LCS may be reported.

10.5. **Laboratory Control Sample Recovery For BOD/CBOD:** Determine the 5 day 20°C BOD of a standard glucose-glutamic acid solution with each analytical batch. If the %R of the LCS result is not within the control limits of 82% to 118%, all analytical batch results must be qualified as directed in SS06 Appendix B, Section 8.

11. REQUIREMENTS FOR INSTRUMENT AND METHOD DETECTION LIMITS

11.1. **General:** Instrument and Method Detection Limits (IDLs/MDLs) shall be determined for each parameter and each instrument used within 30 days prior to and before the start of any contract analyses. MDL determinations are not required for pH and conductivity methods. The laboratory shall determine IDLs for automated and semi-automated instruments; and MDLs for manual

analyses at least semiannually. The IDL/MDL shall be less than or equal to the RDL levels specified in SS06 Table C1. Manual analyses are those in which no autosampler is used, no automatic instrumentation is used for processing samples and reagents, the analyst reads each sample measurement directly from the instrument, and computes each analytical result.

- 11.2. **SW-846 Methods:** For the analysis of samples with Line Item Codes specifying SW-846 as the Approved Methods Source, MDLs must be determined as specified in SW-846.
- 11.3. **EPA-600 Methods:** The IDLs for automated and semi-automated instruments are determined from three sets of analyses of a standard solution (each analyte in reagent water) at a concentration of two to five times the previously-determined or expected IDL. Each set shall consist of the analysis of seven consecutive replicates and be completed on nonconsecutive days. The IDL shall be determined by multiplying by three the average of the standard deviations (σ_{n-1}) from the three standard deviations of the three analysis sets. The MDLs for manual analysis methods are determined by multiplying by three the standard deviation (σ_{n-1}) obtained on the consecutive analyses of seven replicates of a standard solution (each analyte in reagent water) at a concentration of two to five times the previously-determined or expected MDL. Each measurement shall be performed as though it were a separate analytical sample (i.e., each measurement shall be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs/MDLs shall be determined and reported for each parameter with the same number of significant figures as the RDL given in Exhibit C Table C1. Conductivity and pH do not require MDL determinations.
- 11.4. **Automatic or Semiautomatic Instruments:** The semiannual determined IDL for an automatic or semi-automatic instrument shall always be used as the IDL for that instrument during the following six months. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument shall be redetermined and the results submitted for use as the established IDL for that instrument for the following six months.
- 11.5. **Reporting:** IDLs shall be reported for each instrument and submitted with each data package (See SS06 Appendix B Form 3). If multiple instruments are used for the analysis of a parameter within an RIN, the highest IDL for the parameter shall be used for reporting concentration values for that RIN.
- 11.6. **IDL/MDL vs RDL:** The IDL/MDL for each parameter shall be less than or equal to the RDL. An exception is granted if the parameter concentration in the samples is greater than or equal to five times the reported IDL/MDL.
- 11.7. **Raw Data:** All raw data for IDL/MDL measurements, including calculations and determined IDLs/MDLs shall be maintained and documented as specified in SS06 Exhibit B for inclusion in the Supporting Documentation Package.

12. TABLE D1, MINIMUM QC REQUIREMENTS SUMMARY

Table D1 summarizes QC requirements of SS06 and base methods in table form. Base method requirements and requirements of SS06 Exhibit D Sections 1 through 11 shall supersede Table D2 in case of conflict. If instructions in Table D2 should conflict with base method requirements or requirements of SS06 Exhibit D Sections 1 through 11, base methods and SS06 instructions shall supersede Table D2.

TABLE D1 MINIMUM WQP REQUIREMENTS

Instrument Calibration

Method Types	Analytical Criteria	Frequency Criteria	Comments	Documentation Required
Colorimetric/Spectrophotometric Ion Chromatography Infrared Spectrometry TOX Potentiometric, ISE Turbidimetric	One blank and at least 3 standards must be used to calibrate the instrument. The correlation coefficient must be greater than or equal to 0.995. For instruments that cannot produce an internal 4-point curve, determine an external calibration curve.	Instruments must be calibrated daily or once every 24 hours <u>and</u> each time the instrument is set up. The IC shall be recalibrated when calibration verification is not acceptable or a change is made effecting the method	For all instruments, one standard used in the calibration should be at the RDL, one standard near the mid-point of the calibration curve, and one standard at the upper limit of the calibration curve.	Raw data documentation must meet requirements of SS06 Exhibit B.
Potentiometric, pH Meter only	Two point calibration bracketing samples for pH greater than 4 and less than 10. The difference between the observed and standard values must be within ± 0.1 S.U	Instruments must be calibrated daily or once every 24 hours, each time the instrument is set up and whenever verification is not acceptable.	The samples and standards must be at room temperature unless the system performs temperature compensation.	Raw data documentation must meet requirements of SS06 Exhibit B.
Potentiometric, Conductivity only	Standardize with 0.0100M KCl at 25°C. The difference between the standard and observed values must not be more than 1%, or the conductivity cell cleaned and recalibrated.	Instruments must be calibrated daily or once every 24 hours, each time the instrument is set up and whenever verification is not acceptable.	The samples and standards must be at room temperature unless the system performs temperature compensation.	Raw data documentation must meet requirements of SS06 Exhibit B.
Titrimetric	Titrant shall be Standardized, pH electrode shall be calibrated with pH 7 and 4 buffers for Alkalinity	Standardize titrant every month, Calibrate pH electrode with each analytical batch	The samples and standards must be at room temperature unless the system performs temperature compensation.	Raw data documentation must meet requirements of SS06 Exhibit B.
Potentiometric, DO (BOD/CBOD)	Calibrate the instrument per the instruments manufacture's instructions.	Calibrate daily or once every 24 hours, each time the instrument is set up.		Raw data documentation must meet requirements of SS06 Exhibit B.

TABLE D1 MINIMUM WQP REQUIREMENTS (continued)

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

Method Types	Analytical Criteria	Frequency Criteria	Comments	Documentation Required
Colorimetric/Spectrophotometric Ion Chromatography Titrimetric TOX Infrared Spectrometry Potentiometric, ISE Turbidimetric	Percent recoveries must be within 85% to 115% of the expected value. If response or retention times for any IC analyte varies from calibration standards by more than 10%, recalibrate.	The ICV must be analyzed immediately after instrument calibration(s). The CCV must be analyzed after every 20 measurements and at the end of each analytical run prior to each CCB.	If %R is outside control limits, analysis must be stopped, the problem corrected, the instrument recalibrated, and the calibration verified.	Raw data documentation must meet requirements of SS06 Exhibit B. Result summaries are reported in WQP QC Summary Report Form 2.
Potentiometric, pH only	The difference between the observed and standard values must be within ± 0.1 S.U	The ICV must be analyzed immediately after instrument calibration(s). The CCV must be analyzed after every 20 measurements and at the end of each analytical run prior to each CCB.	The standards must be at room temperature unless the system performs temperature compensation. If ICV or CCV is outside the control limits, the analysis must be stopped, the problem corrected, the instrument recalibrated,, the calibration verified and samples reanalyzed to the last in-control verification.	Raw data documentation must meet requirements of SS06 Exhibit B. Result summaries are reported in WQP QC Summary Report Form 2.
Potentiometric, conductivity only	For the ICV & CCV at least 1 standard KCl solution, other than the 0.01M KCl standardization solution, near the sample conductivity shall be analyzed. When possible bracket the sample conductivity. If the %R is not within 95% to 105%, recalibrate.	The ICV must be analyzed immediately after instrument calibration(s). The CCV must be analyzed after every 20 measurements and at the end of each analytical run prior to each CCB.	The standards must be at room temperature unless the system performs temperature compensation. If ICV or CCV is outside the control limits, the analysis must be stopped, the problem corrected, the instrument recalibrated,, the calibration verified and samples reanalyzed to the last in-control verification.	Raw data documentation must meet requirements of SS06 Exhibit B. Result summaries are reported in WQP QC Summary Report Form 2.
Gravimetric	Standardization Verification shall be performed with traceable Check weights per GR01 Exhibit E Section 5.	The standard verification shall be performed before and after measurement of samples each day.	The difference between the observed and standard values shall be less than 0.0010 grams.	Raw data documentation must meet requirements of SS06 Exhibit B. Result summaries are reported in WQP QC Summary Report Form 2.

TABLE D1 MINIMUM WQP REQUIREMENTS (continued)***Initial and Continuing Calibration Blanks (ICB & CCBS)***

Method Types	Analytical Criteria	Frequency Criteria	Comments	Documentation Required
Colorimetric/Spectrophotometric Ion Chromatography Titrimetric TOX Infrared Spectrometry Potentiometric, ISE Turbidimetric	The absolute value of the blank reading must be less than or equal to the RDL.	An ICB is analyzed immediately following the ICV. CCBs are analyzed every 20 measurements immediately following the CCV and at the end and at the end of each analytical run..	If the ICB or CCB reading is outside of control limits, the analysis must be stopped, the problem corrected, the calibration verified, and samples analyzed since the last in-control calibration verification must be reanalyzed.	Raw data documentation must meet requirements of SS06 Exhibit B. Result summaries are reported in WQP QC Summary Report Form 3.

Preparation Blanks/Method Blanks (PB)

Method Types	Analytical Criteria	Frequency Criteria	Comments	Documentation Required
Colorimetric/Spectrophotometric Ion Chromatography TOX Infrared Spectrometry Titrimetric Gravimetric Turbidimetric Potentiometric, DO, (BOD/CBOD) Potentiometric, ISE	The BOD/CBOD dilution water-seed blank uptake shall be prepared and reported. No control limit is applicable for the HACH Method. The absolute value of all other preparation blank readings must be less than or equal to the RDL.	A preparation blank must be prepared and analyzed with each analytical batch for samples requiring preparation before analysis.	If the PB reading is outside of control limits, all samples in the analytical batch with analyzed concentrations of the affected analyte less than ten times the analyzed concentration of the PB must be reprepared and reanalyzed.	Raw data documentation must meet requirements of SS06 Exhibit B. Result summaries are reported in WQP QC Summary Report Form 3.

Laboratory Control Sample (LCS)

Method Types	Analytical Criteria	Frequency Criteria	Comments	Documentation Required
Colorimetric/Spectrophotometric Ion Chromatography TOX Infrared Spectrometry Titrimetric Gravimetric Turbidimetric Potentiometric, DO, (BOD/CBOD) Potentiometric, pH, ISE, Cond.	Measured LCS values for pH must be within 0.1 S.U. of the expected value. Measured LCS values for Conductivity must be within 5% of the expected value. Measured LCS values for BOD/CBOD must be within 18% of the expected value. Percent recoveries (%R) for all other analyses must be within 80% to 120% of the expected value for aqueous control samples.	An LCS must be prepared and analyzed with each analytical batch.	If the LCS reading is outside of control limits, all samples in the analytical batch must be reanalyzed after preparation of another analytical batch, if necessary.	Raw data documentation must meet requirements of SS06 Exhibit B. Result summaries are reported in WQP QC Summary Report Form 6.

TABLE D1 MINIMUM WQP REQUIREMENTS (continued)

Matrix Spikes

Method Types	Analytical Criteria	Frequency Criteria	Comments	Documentation Required
Colorimetric/Spectrophotometric Ion Chromatography TOX Infrared Spectrometry titrimetric Potentiometric, ISE Turbidimetric	Percent recoveries (%R) must be within 75% to 125% of the expected value if the sample analyte concentration is less than 4 times the concentration of spike added.	One matrix spike must be prepared and analyzed with each analytical batch and each sample type, whichever is more frequent.	Samples identified as field blanks must not be used for matrix spike analysis. If %R is outside control limits all samples associated with the matrix spike sample shall be reanalyzed with the problem corrected or have the results flagged.	Raw data documentation must meet requirements of SS06 Exhibit B. Result summaries are reported in WQP QC Summary Report Form 4.

Laboratory Duplicates

Method Types	Analytical Criteria	Frequency Criteria	Comments	Documentation Required
Colorimetric/Spectrophotometric Ion Chromatography TOX Infrared Spectrometry Titrimetric Gravimetric Turbidimetric Potentiometric, Cond., ISE, pH Potentiometric, DO, (BOD/CBOD)	Sample and duplicate values for pH must be within 0.1 S.U. and the RPD for Conductivity must be < 5%. For other parameters, the Relative percent difference (RPD) must be less than 20% for sample concentrations greater than five times the RDL or the absolute value of the difference between sample and duplicate results must be less than the RDL for sample concentrations less than 5 times the RDL.	One laboratory duplicate must be prepared and analyzed with each analytical batch and each sample type, whichever is more frequent.	Samples identified as field blanks must not be used for laboratory duplicate analysis. If agreement between sample and duplicate samples is outside control limits all samples associated with the duplicate shall be reanalyzed with the problem corrected or have the results flagged.	Raw data documentation must meet requirements of SS06 Exhibit B. Result summaries are reported in WQP QC Summary Report Form 5.

13. TABLE D2, CODES FOR LABELING DATA

Table D2 contains suggested data labeling codes. Use of other codes will require clarification.

TABLE D2 CODES FOR LABELING DATA

Description	Prefix	Suffix	Root
Site Samples	(1)	(1)	Site or Lab sample identifier
Sample not part of RIN	ZZ	ZZ	ZZ
Laboratory Duplicate	(1)	D	Site or Lab sample identifier
Matrix Spike	(1)	S	Site or Lab sample identifier
Post Preparation Spike (used only if applicable for the Analysis Type)	(1)	A	Site or Lab sample identifier
Calibration/Standardization Standards	S	(1)	Standard Tracking Number ⁽²⁾
Initial Calibration Verification	ICV	(1)	Standard Tracking Number ⁽²⁾
Initial Calibration Blank Verification	ICB	(1)	Standard Tracking Number ⁽²⁾
Continuing Calibration Verification	CCV	(1)	Standard Tracking Number ⁽²⁾
Continuing Calibration Blank	CCB	(1)	Standard Tracking Number ⁽²⁾
Laboratory Control Sample for Water	LCSW	(1)	(3)
Preparation Blank for Water	PBW	(1)	(3)

(1) No format or entry requirements have been set for this item.

(2) This is the Secondary Standard Tracking Identifier specified in GR01 Exhibit E Section 8. If the Secondary Standard Tracking Identifier begins with the indicated prefix, it is not necessary to repeat that portion of the identifier.

(3) Must meet requirements of SS06 Exhibit F Section 4 requirements for linking analytical batch raw data.

14. REQUIREMENTS FOR HACH BOD/CBOD GRAPHICAL CALCULATION METHOD

The following guidelines shall be followed for determining and calculating CBOD and BOD results by the HACH Graphical Calculation Method for LICs SS06*006 and SS06*008.

14.1. Five sample dilutions for each Site sample and LCS shall be processed. Initial DO values of diluted samples are not required.

14.2. The Sample DO value (S) of the undiluted sample shall be used for the S value in the following formula when calculating BOD or CBOD results:

$$\text{BOD/CBOD (mg/L)} = 300(\text{Slope}) - Y \text{ intercept} + S$$

14.3. Slope Value

14.3.1. The Slope value for calculating BOD/CBOD (mg/L) in the above formula shall be determined from a linear plot of the diluted sample volumes processed and the final diluted sample DO values after incubation.

14.3.2. The linear plot should have a correlation coefficient (r^2) of at least 0.95.

14.3.3. Analytical judgment shall be used to eliminate data points that are not consistent with the other data points to obtain an acceptable linear plot of the data. . For example, if there are four points on a straight line and one point differs from the general line of the others, it should be discarded. Also, if three or more data points fall on a straight line, they could all be used even though one does not deplete more than 2 mg/L oxygen or leave more than 1 mg/L. Documentation shall be provided in the raw data regarding any outlier that is discarded.

14.3.4. Documentation shall be provided in the SDP Narrative for any results determined from a plot with less than three data points or a linear plot with a correlation coefficient (r^2) of less than 0.95.

14.4. Y Intercept Value

14.4.1. The Y intercept of the plot should agree within ± 0.5 mg/L of the DO value of the dilution water and seed blank after incubation (final blank DO value). Alternately, the Y intercept cannot be above the DO value of the saturated dilution water minus the seed correction. . Documentation shall be provided in the raw data regarding any outlier that is discarded.

14.4.2. Analytical judgment shall also be used when determining a linear plot based upon the Y intercept. Documentation shall be provided in the raw data regarding any outlier that is discarded.

14.4.3. Documentation shall be provided in the SDP Narrative for any results determined from a plot with a Y intercept that is not within ± 0.5 mg/L of the DO value of the dilution water and seed blank after incubation (final blank DO value).

14.5. **Additional Requirements:** Additional requirements are given in Sections 1 through 13 of this SS06 Exhibit D.

EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

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QUALITY ASSURANCE /QUALITY CONTROL REQUIREMENTS

1. INTRODUCTION

The purpose of this Exhibit is to describe the minimum QA/QC operations necessary to satisfy the analytical requirements associated with the determination of the target Water Quality Parameters analysis. These operations and those in the General Laboratory Requirements Module GR01 are designed to ensure the generation of comparable data from all laboratories. These requirements do not release the Laboratory from maintaining its own QC checks on method and instrument performance.

2. QUALITY ASSURANCE PLAN

Requirements for the quality assurance plan are specified in GR01 Exhibit E.

3. ANALYTICAL STANDARDS AND REAGENTS REQUIREMENTS

- 3.1. **Multi-parameter Standards:** Primary standards used for multi-parameter secondary standards shall include additional certification statements or tables quantifying or verifying the absence of all other parameters included in the multi-element standards.
- 3.2. **General Requirements:** Additional requirements for the analytical standards and reagents are specified in Exhibit E of the General Laboratory Requirements Module, GR01.

4. METHOD SPECIFIC QC REQUIREMENTS

- 4.1. **Default Protocols:** Where base methods do not require specific QC protocols, laboratory SOPs shall specify requirements which meet or exceed parallel requirements found in this SOW.
- 4.2. **General Requirements:** Other method specific QC requirements have been included in SS06 Exhibit D.

5. MEASURING AND TESTING EQUIPMENT REQUIREMENTS

Requirements for measuring and testing equipment are specified in GR01.

6. DATA MANAGEMENT

Requirements for site data management are specified in GR01.

7. LABORATORY EVALUATION SAMPLES

- 7.1. **Inter-laboratory:** The Laboratory shall participate in an inter-laboratory comparison study on a quarterly basis that includes the following parameters at a minimum: total alkalinity, ammonia as N, BOD, chloride, total cyanide, fluoride, hardness, total nitrate/nitrite as N, pH, total phosphate, NVSS, TDS, TSS, and sulfate.
- 7.2. **Regulatory PE Samples:** All NPDES, EPA and State of Colorado PE samples shall be analyzed when submitted.
- 7.3. **Other:** Requirements for laboratory evaluation samples are specified in the General Laboratory Requirements Module, GR01.

8. ON-SITE LABORATORY EVALUATIONS

Requirements for on-site laboratory evaluations are specified in the General Laboratory Requirements Module, GR01.

9. PERFORMANCE CRITERIA

Laboratory performance will be continually assessed by the CTR. Performance areas will include those outlined in the General Laboratory Requirements Module GR01 Exhibit E, Section 13, and those outlined below:

- 9.1. **Holding Time:** Hold-time shall be used as a performance measure for all applicable water quality parameters. The Site will monitor the Laboratory for compliance to holding time requirements as described in SS06 Exhibit D, Section 3.

10. CERTIFICATIONS AND APPROVALS

Laboratories are encouraged to provide proof of certifications or approvals for any programs in which the Laboratory is participating. Proof of laboratory certifications (or approvals) delivered to the CTR according to the schedule in GR01 Exhibit B Table B2 may increase the potential sample load for Laboratories providing this proof.

- 10.1. Laboratories providing analytical services under Module SS06 are required to have the State of Colorado or USEPA certifications or approvals related to the determination of water quality parameters for samples related to US EPA National Pollutant Discharge Elimination System (NPDES) permits.

EXHIBIT F

EVIDENTIARY REQUIREMENTS

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EVIDENTIARY REQUIREMENTS

1. INTRODUCTION

The purpose of this Exhibit is to describe the evidentiary requirements that must be followed for the preparation and analysis of Site samples for target Water Quality Parameters under this subcontract.

2. SAMPLE CHAIN-OF-CUSTODY

Requirements for sample chain-of-custody are specified in GR01.

3. SAMPLE RECEIVING REQUIREMENTS

Requirements for sample receiving are specified in GR01.

4. DOCUMENT CONTROL PROCEDURES

4.1. **General Requirements:** General requirements for document control are specified in GR01.

4.2. **Raw Data Labeling Requirements:** Results must be presented with all raw data necessary for the CTR to assess their validity. This verification process involves such processes as recalculating reported values and qualifiers from raw data and tracing all standards to valid reference materials. In order to perform this verification, the following rules must be followed for labeling all raw data submitted in Sample Data Packages and Supporting Documentation Packages (Support Packages). Preparation, instrument, and other raw data must meet the following labeling requirements:

- 4.2.1. Sample data must be labeled with the Site Sample Identifiers or with the lab identifiers cross-referenced to the Site Sample Identifiers on the Sample Data Package Cover Page.
- 4.2.2. All data generated by M&TE must be labeled with the unique M&TE identifier.
- 4.2.3. Data labels must clearly identify samples designated as duplicates, spiked samples, controls, and blanks. The data labeling codes specified in SS06 Exhibit D Table D2: *Codes for Labeling Data* may be used without clarification. Use of other identification schemes will require all codes to be clarified in a description table on each affected Section Cover Sheet.
- 4.2.4. The identification scheme used (whether using SS06 Table D2 or alternate codes) must provide an unequivocal and unique link between all samples and QC samples (lab duplicate, spiked sample, laboratory control sample, preparation blank) prepared as an analytical batch. For example, a preparation blank labeled as 'PBW' appended by the Analytical Batch Identifier (see GR01 Exhibit G) would provide this link.
- 4.2.5. All standards referenced in raw data must be identified by the unique identifier assigned as required in GR01 Exhibit E Section 6.
- 4.2.6. All numerical data shall be accompanied by applicable units.

5. STANDARD OPERATING PROCEDURES

5.1. **General Requirements:** Requirements for written SOPs are specified in GR01.

5.2. **Required Procedures:** For each analysis technique used by the Laboratory, each of the following operations shall be included in one or more SOPs:

- Instrument Calibration
- Initial Calibration Verification and Continuing Calibration Verification
- Calibration Blanks Verifications.
- Preparation Blanks
- Spike Sample Analysis
- Laboratory Duplicate Sample Analysis
- Laboratory Control Sample Analysis
- Performance Evaluation (PE) Sample Studies
- Method Detection Limit (MDL) Determination

EXHIBIT G

GLOSSARY OF TERMS AND ACRONYMS

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GLOSSARY OF TERMS AND ACRONYMS

1. INTRODUCTION

The glossary of terms and acronyms contained in parameter-specific analytical module(s) supplement those found in the General Laboratory Requirements Module, GR01. Any definitions provided in this Exhibit, however, shall supersede the definitions provided in GR01 in cases of conflicting definitions.

2. GLOSSARY OF TERMS

Refer to General Requirements Module GR01, Exhibit G for additional terms.

ABSORBANCE - A measure of the decrease in incident light passing through a sample into the detector.

ANALYSIS DATE/TIME - The date and time (24-hour clock) of the analytical measurement of the sample, standard, or blank by the analysis system.

ANALYSIS LOGSHEET/BENCHSHEET - A form with lab and analysis method identifiers; calibration information; sample identifiers; sample volumes, weights, dilution factors and measurement data; standard traceability information; date/times of analysis; and signatures of analyst and reviewer.

ANALYTICAL (ANALYSIS) SPIKE - A post-digestion spike to be prepared prior to analysis by adding a known quantity of the analyte to an aliquot of the prepared sample. The unspiked sample aliquot must compensate for any volume change in the spike samples by addition of ASTM Type II water to the unspiked sample aliquot. The volume of the spiking solution added must not exceed 10% of the analytical sample volume.

AUTOZERO - Zeroing the instrument at the proper wavelength. It is equivalent to running a standard blank with the absorbance set at zero.

CALIBRATION BLANK - A blank solution containing all of the reagents and in the same concentration as those used in the analytical sample preparation. This blank is subjected to the preparation method, if applicable but is produced synthetically.

CALIBRATION CURVE - For WQP, see SS06 Exhibit D Section 5

CALIBRATION/STANDARDIZATION DATE/TIME - The date and time (24-hour clock) that the analytical system calibration/standardization was completed.

CALIBRATION STANDARDS - A series of known standard solutions used by the analyst for calibration of the instrument (i.e. preparation of the analytical curve). For WQP, the solutions may be subjected to the preparation method but contain the same matrix as the sample preparations to be analyzed.

CONTROL LIMITS - A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

C Qualifiers - Concentration Qualifiers for WQP are U and B. A *U* is used to indicate a result that was not detected with the MDL value reported. A *B* is used to indicate a result that was less than the RDL, but greater than or equal to the MDL.

INITIAL/CONTINUING CALIBRATION VERIFICATION (ICV/CCV) - A single element or multi-element standard solution prepared by the analyst to be used to verify the stability of the instrument calibration with time and the instrument performance during the analysis of samples. The initial/continuing calibration verification solution(s) is either the same solution or two different solutions that are independent of the calibrations standards source. However, all analyte elements being measured by the particular system must be represented in this standard and the standard must have the same matrix as the samples. The initial/continuing calibration solution(s) should have a concentration near the mid-point of the analytical calibration curve. The initial calibration verification solution must be analyzed immediately after calibration and prior to analysis of any samples. The continuing calibration verification solution must be analyzed every 20 analytical samples and after the last analytical sample to verify the calibration of the analytical system.

PERCENT RECOVERY (%R) - For ICV/CCV standards see SS06 Appendix B Sections 4.4 and 4.7. For sample-spike recovery, see SS06 Appendix B Section 6.6. For Laboratory Control Sample recovery, see SS06 Appendix B Section 8.4.

PREPARATION DATE/TIME - The date and time (24-hour clock) of the preparation of the sample, standard, or blank was started.

PREPARATION LOGSHEET/BENCHSHEET - A form with lab and analysis method identifiers; sample identifiers; sample volumes, weights, and dilution factors; standard traceability information; date/times of preparation; and signatures of analyst and reviewer.

RELATIVE PERCENT DIFFERENCE (RPD) - see SS06 Appendix B Section 7.5.1

Q Qualifier - Quality Qualifiers for WQPs are N and *. An 'N' is used to indicate the %R for a Spiked Sample result not within the control limits when the sample concentration is less than or equal to four times the spike amount. Also, an 'N' is used to qualify sample results that have a matrix similar to a spiked sample when the %R is not within the control limits. A '*' is used to indicate a duplicate result which is not within control limits. Also, an '*' is used to qualify sample results that have a matrix similar to a duplicate sample result in which the agreement is not within the control limits

3. ACRONYMS

Refer to General Requirements Module GR01, Exhibit G for all applicable acronyms.

B	A concentration (C) qualifier defined in SS06 Appendix B
C Qualifier	Concentration Qualifier
DO	Dissolved Oxygen
ISE	Ion Selective Electrode
ICB/CCB	Initial Calibration Blank/Continuing Calibration Blank
ICV/CCV	Initial Calibration Verification/Continuing Calibration Verification
N	A quality (Q) qualifier defined in SS06 Appendix B

NR	Not Required
%R	Percent Recovery
SA	Spike Added
SR	Spike Result
SSR	Spike Sample Result
S.U.	Standard Units
Q Qualifier	Quality Qualifier
U	A concentration (C) qualifier defined in SS06 Appendix B
UV	Ultraviolet
*	A quality (Q) qualifier defined in SS06 Appendix B

EXHIBIT H

REFERENCES

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REFERENCES

1. REFERENCES

1.1. EPA-600

'Methods for Chemical Analysis of Water and Wastes,' *EPA-600/4-79-020*, Revised March 1983 and 1979 where applicable, Environmental Protection Agency, Environmental Monitoring Systems Laboratory-Cincinnati (EMSL-CI)

1.2. SW-846

'Test Methods for Evaluating Solid Waste,' *SW-846*, Third Edition, November 1986, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response.

1.3. Standard Methods

'Standard Methods for the Examination of Water and Wastewater,' 18th Edition, 1992, American Public Health Association (APHA), New York, NY.

1.4. Other References Cited

'Standard Specifications for Reagent Water,' ASTM D 1198, American Society for Testing and Materials (ASTM).

'Methods for Water Analysis,' Volume 11.01 and 11.02, Water 1 and 2, 1993, American Society for Testing and Materials (ASTM) (or latest revision)

'Laboratory Theory And Methods For Sediment Analysis, Chapter C1 of Techniques of Water-Resources Investigations of the United States Geological Survey,' 1969; Harold P. Guy, U.S. Geological Survey.

'Quality-Assurance Plan For The Analysis Of Fluvial Sediment By Laboratories Of The U.S. Geological Survey,' U. S. Geological Survey Open-File Report 91-467, Wilbur J. Matthes, Jr., Clyde J. Sholar, and John R. George, U. S. Geological Survey.

'Standard Methods for the Examination of Water and Wastewater,' 19th Edition, 1995, American Public Health Association (APHA), New York, NY.

A P P E N D I X A

Data Review Checklist

SS06 SAMPLE DATA PACKAGE

Data Review Checklist

SS06 Sample Data Package

1. SAMPLE DATA PACKAGE COVER PAGE	Reply	Ö	C#
a) The laboratory name, code, subcontract number, RIN, Site sample numbers, Line Item codes (LIC-analyses), sample matrix, and report dates are accurately recorded.			
b) All Site sample identifications are cross-referenced with all lab identifications.			
c) The verbatim compliance and authorization statement is present with the dated signature of the Laboratory Manager or designee.			
d) Any problems with the receipt are explained (e.g., broken containers, incorrect COC documentation, etc.)			

2. TABLE OF CONTENTS	Reply	Ö	C#
a) The Table of Contents is included and contains all Sample Data Package Deliverable Section Titles with their beginning page numbers.			

3. DATA REVIEW CHECKLIST - SS06 SAMPLE DATA PACKAGE	Reply	Ö	C#
a) The SS06 DRC is present and in strict conformance with the formatting and content of the form contained in the current version of the SS06 Appendix A. All discrepancies were identified and documented, accordingly.			
b) All DRC Reply blocks are completed with either a “Y”, “N” or “N/A”.			
c) All DRC Reply blocks completed with an “N” are explained in the Narrative.			
d) The DRC footer is completed for each page; the laboratory manager or designee signed and dated the DRC.			
e) All SDP deliverable sections appear in the SDP in order by deliverable section number.			
f) Only one SDP is submitted for each SS05 and RIN request.			N/A
g) All components of the SDP deliverables contain original documents where possible.			Φ
h) There is no inclusion of required items in the SDP by reference to another SDP.			Φ
i) Site samples are exclusively used for sample matrix QC.			Φ
j) Site and non Site samples are not reported together in any way.			Φ
k) The complete sample data package is single sided and consecutively paginated.			

4. CHAIN-OF-CUSTODY, HOLDING TIMES, AND SAMPLE PRESERVATION	Reply	Ö	C#
a) The continuity of each sample's custody is evidenced by the chain of the date, time and signatures of each transaction from sample collection to receipt by the laboratory.			
b) If the continuity was corrupted, documentation of correspondence with the CTR is included.			
c) All samples are identified on the COC with the corresponding Line Item Codes (analyses).			
d) The pH of each sample and the shipping container temperature are recorded, where applicable; Preservation was consistent with PSA Module.			
e) Any conflicting, incorrect, or missing information are identified and documented, and there is documentation of the resolution.			
f) Analytical and preparation holding times were met for all sample analyses.			
g) Following sample receipt by the lab, samples were properly preserved and were stored at the appropriate temperature, if required. Internal COCs are included.			

RIN: _____ Lab Name: _____ Initials: _____

Analytical Batch Identification No.(s) : _____

Data Review Checklist

SS06 Sample Data Package

5. NARRATIVE	Reply	Ö	C#
a) Contains a synopsis of the analytical and preparation methods; identifies base methods and any deviations of the base methods.			
b) Contains a description of the samples; describes samples which are of a similar matrix.			
c) Contains synopsis of Analytical Batch QC assessment. All anomalies, deficiencies, interferences, reanalyses, and deviations from approved SOPs related to the analysis are explained. Contains a QC assessment for the RIN which includes a discussion of all items with an "N" reply on the SSO6 DRC.			
1. statement about N, E, and * flags			
2. statement if dilutions required, and all dilutions explained			
3. statement saying whether the MDL meets the RDL for each parameter.			
4. statement saying whether all calibration verifications were or were not met			
5. statement saying whether all blank values were less than the RDL for all parameters			
6. statement saying whether duplicate sample analysis was performed per SS06 and the results were or were not within acceptable limits of SS06			
7. statement saying whether matrix spike sample analysis was performed per SS06 and the results were or were not within acceptable limits of SS06			
8. statement saying whether laboratory control sample analysis was performed per SS06 and the results were or were not within acceptable limits of SS06			
d) Samples requiring reanalysis are identified with the reason for reanalysis, the original and reanalysis Analytical Batch Identification Numbers. A synopsis of the reanalysis Analytical Batch QC assessment is included.			
e) For any deviations that required CTR approval, the correspondence and approval are documented.			
f) All holding-time compliances and violations are described in Narrative.			

6. SAMPLE AND QC RESULTS SUMMARY	Reply	Ö	C#
The Sample and QC Results Summary Package is present and all pages are labeled with the Lab Code and the RIN. The Sample and QC Results Summary Package includes:			
a) Form 1s (Sample Results) are present for each sample in the RIN for this PSA Module and each includes:			
1. one and only one result for each requested analyte.			
2. correct results reported without blank correction in the correct units for each requested analyte.			
3. results for detected analytes factored by all dilutions.			
4. results for non-detected analytes reported as MDLs factored by all dilutions.			
5. results reported to the correct number of significant figures.			
6. C qualifiers entered correctly for each analyte.			
7. Q qualifiers entered for all samples associated with QC samples receiving qualifiers.			
8. the (MDL * dilution factor) for non-detected analyte is ≤ the specified RDLs.			
b) Form 2s (Calibration Verification) are present and each includes:			
1. all ICV/CCV results and %R values are reported appropriately for all applicable parameters.			
2. results for an ICV from the beginning of the run, and a CCV at the end of the run.			Φ
3. CCV results reported such that no more than 20 solutions (except CCB or ICB) were analyzed between analysis of ICVs or CCVs.			Φ

RIN: _____ Lab Name: _____ Initials: _____

Analytical Batch Identification No.(s) : _____

Data Review Checklist

SS06 Sample Data Package

(continued)	Reply	√	C#
4. all ICV and CCV results are within limits.			
5. if the preceding item was marked "N," no sample data were reported when results were not within limits.			
6. at least the minimum number of standards at required levels and frequency prescribed for each method were used to establish the initial calibration, if applicable			Φ
7. The correlation coefficients of the calibration curves were > 0.995 for all analyses.			Φ
8. The calibration and ICV/CCV standards were from independent sources.			Φ
c) Form 3s (Verification and Preparation Blanks) are present for each method with the respective MDLs and each includes:			
1. all ICB/CCB results for all applicable parameters; results reported without blank subtraction and reported to MDLs.			
2. results for an ICB from the beginning of the run, and a CCB at the end of the run.			Φ
3. CCB results reported such that no more than 20 solutions (except CCV) were analyzed between analysis of ICBs or CCBs.			Φ
4. the absolute value of all ICB and CCB results are less than specified MDLs.			
5. if the preceding item was marked "N," and the blank results are >RDL, then no sample data were reported from the non-conforming analytical batch.			
6. PB results are reported for all applicable parameters for each analytical batch in the RIN; results reported without blank subtraction and reported to MDLs.			
7. the absolute values of analyte concentrations in PBs for all applicable parameters are less than specified MDLs.			
8. if the preceding item was marked "N," and the blank results are >RDL, then only samples containing >5(PB) concentration are reported for that analyte from the non-conforming analytical batch.			
d) Form 4As (Matrix Spike) are present and each includes:			
1. one Form 4A for each matrix, waste type, and analytical batch (max. of 20 samples).			
2. control limits for %R assigned according to SS06 Appendix B			
3. spikes reported and completed for all applicable parameters according to SS06 Exhibit D & Appendix B			
4. "N" flags present in the Q column if %R is outside limits according to SS06 Appendix B			
e) Form 4Bs (Post Digestion Spike) are present for each matrix and analyte receiving an N flag on Form 4A.			
f) Form 5s (Duplicate) are present and each includes:			
1. one Form 5 for each matrix, waste type, and analytical batch (max. of 20 samples).			
2. control limits for RPD assigned according to SS06 Appendix B.			
3. RPD reported and calculated according to SS06 Appendix B.			
4. "*" flag present in the Q column if RPD is outside limits according to SS06 Appendix B.			
g) Form 6s (Laboratory Control Sample) are present and each includes:			
1. A Form 6 or LCS result for each analytical batch is present; results for all requested parameter.			
2. LCS results for all requested parameters and %R values are within the control limits.			
3. "E" flags present in the Q column if %R is outside limits according to SS06 Appendix B.			
h) Form 7s (Sample Holding Time Summary) are present for each WQP.			Φ

RIN: _____ Lab Name: _____ Initials: _____

Analytical Batch Identification No.(s) : _____

Data Review Checklist

SS06 Sample Data Package

7. PREPARATION RAW DATA	Reply	Ö	C#
a) The preparation raw data (benchsheets and/or preparation logs) are included and document the required items as specified in the Preparation Summary Section of SS06, Exhibit B, Section 2.9.3.			Φ
b) Sufficient raw data are included to allow manual calculation of the final sample results and QC sample recoveries.			Φ
c) Samples were prepared using an approved procedure in SS06 Exhibit C Table C1.			

8. STANDARDS SUMMARY	Reply	Ö	C#
a) For primary standards that were diluted and used for LCS, ICV/CCV and any in-house prepared instrument calibration solutions, the required items as specified in the Standards Summary Section of SS06, Exhibit B are included.			Φ
b) All standard identifications are traceable to the primary certificates, which are traceable to NIST, when possible.			Φ
c) All standards and sources were valid (not expired) at the time of use.			Φ

9. INSTRUMENT RAW DATA	Reply	Ö	C#
a) The instrument raw data for the RIN are included and document the required items as specified in SS06, Exhibit B, Section 2.11.2. All data entries were verified as accurate by the reviewer.			Φ
b) The data were reviewed, signed and dated by an area specialist, and found to be acceptable			Φ
c) Sufficient raw data are included to allow manual calculation of the final sample results and QC sample recoveries.			Φ
d) All QC samples were prepared and analyzed in the same manner as the samples in the Analytical Batch, in the same time frame, and using the same instrument and instrument parameters, etc.			Φ

10. ELECTRONIC DATA DELIVERABLE (EDD)	Reply	Ö	C#
a) The EDD accurately reflects the data contained in the Sample Data Package.			
b) The hard copy of the EDD as specified in GR01 Exhibit B, Section 4 is included with the Sample Data Package. The hard copy includes data file name and means of transmittal.			
c) An automated EDD verification check has been performed.			N/A

Shaded areas are for Site use only.

*Respond to each checklist item in the "Reply" column with a Y (yes), N (no), or NA (not applicable).
Complete footer information, including the initials of the laboratory manager or designee on each page.
Refer to Module GR01, Exhibit B, Section 4 for instructions to complete this form.*

I certify that all responses to this checklist accurately reflect the completeness and quality aspects of this sample data package as outlined in GR01 and SS06. Furthermore, I understand that inaccuracies in the completion of this checklist will be considered a nonconformance to Subcontract Requirements as evidenced by the following signature of the laboratory manager or designee.

Print/Typed Name: _____ Title: _____

Signature _____ Date _____

RIN: _____ Lab Name: _____ Initials: _____

Analytical Batch Identification No.(s) : _____

DA-SS06 Version _____

Notes:

Date _____

Notes:

Date _____

Page 5 of 6

COMPLETE VERIFICATION

- ☐ This Sample Data Package requires no further assessment (See attached Data Quality Assessment Report)
- ☐ This Sample Data Package requires validation

Notes:

Data Verifier

Signature

Date

RIN: _____ Lab Name: _____ Initials: _____

Analytical Batch Identification No.(s) : _____

Appendix B

WATER QUALITY PARAMETERS REPORTING FORMS

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WATER QUALITY PARAMETERS REPORTING FORMS

1. INTRODUCTION

SS06 Exhibit B Section 2 requires submittal of a Sample Data Package for compliance to this SOW; Section 6 of this Sample Data Package is called the *Sample and QC Result Summaries*. SS06 Appendix B contains form templates and instructions for producing the *Sample and QC Result Summaries* portion of the Sample Data Package for Water Quality Parameters.

2. GENERAL INSTRUCTIONS

- 2.1. **Compliance to Formats:** Forms submitted in the sample data package must resemble the forms provided in this appendix. This means that the following items shall be as presented on Forms 1 through 6 in SS06 Appendix B:

- 2.1.1. Parameter order
- 2.1.2. Form number
- 2.1.3. Form Title
- 2.1.4. Header information
- 2.1.5. Column headers and order
- 2.1.6. Form footnotes and comments

- 2.2. **Allowed Variances:** Some items do not require strict compliance with supplied formats. Format modifications may vary for the following:

- 2.2.1. Printer font style and size may vary from the supplied formats.
- 2.2.2. Additional information columns may be included on forms, if this information does not affect the intelligibility of required items. Additional information must either be self-explanatory or be accompanied by notes which explain the content of additional information.
- 2.2.3. Forms should be modified to include only requested parameters. In other words, result table rows are required only for requested parameters. If a parameter is not requested, the row for that parameter should be omitted for all forms. If form rows are included for parameters not requested, then *NR* must be entered in the field or cell immediately to the right of the parameter name.
- 2.2.4. Forms 1 through 6 must all contain the same number of parameters. If QC information for a specific analysis type is not required for one or more forms, enter NA in the field or cell immediately to the right of the parameter name.

3. WQP FORM 1, WQP ANALYSIS DATA SHEET

WQP Form 1 contains sample analysis results and qualifiers for requested parameters. A complete Form 1 shall be submitted for each sample reported.

- 3.1. **Parameter ID:** The *Parameter ID* column shall contain the parameter identifier for each requested analyte as listed in SS06 Exhibit C Table C1.

- 3.2. **Parameter Name:** The *Parameter Name* column shall contain the name of each requested parameter identified exactly as exactly as presented in SS06 Appendix B Form 1.
- 3.3. **Result:** Complete the column labeled *Result* for each requested parameter as follows:
- 3.3.1. Enter the value of the measured result, corrected for any dilutions, if the measured concentration is greater than or equal to the MDL.
 - 3.3.2. Enter the MDL for the parameter, corrected for any dilutions, if the measured concentration is less than the MDL.
 - 3.3.3. Report results in the units listed in the *Units* column of SS06 Appendix B, Form 1.
 - 3.3.4. Report all results which exceed the MDL by a factor of ten or more to three significant figures. Results between the MDL and ten times the MDL may be reported to two or three significant figures.
- 3.4. **Qualifiers, C:** Complete the column labeled *C Qualifiers* for each requested parameter as follows:
- 3.4.1. Enter a 'U' if the reported value was less than the MDL.
 - 3.4.2. Enter a 'B' if the result is greater than or equal to the MDL, but less than the RDL.
- 3.5. **Qualifiers, Q:** Complete the column labeled *Q Qualifiers* for each requested parameter as follows:
- 3.5.1. Enter an 'N' if a spike recovery qualifier is to be assigned to an associated sample-spike as defined in SS06 Appendix B instructions for completion of Form 4.
 - 3.5.2. Enter an '*' if a duplicate agreement qualifier is to be assigned to an associated duplicate sample as defined in SS06 Appendix B instructions for completion of Form 5.
 - 3.5.3. For BOD and CBOD results only, enter an 'E' for all samples in the analytical batch if an LCS qualifier is to be assigned as defined in SS06 Appendix B instructions for completion of Form 6.
 - 3.5.4. Leave blank if neither spike recovery, duplicate agreement, nor BOD LCS qualifiers are required.
- 3.6. **Units:** The column labeled *Units* must contain unit designators exactly as presented in SS06 Appendix B Form 1.

4. WQP FORM 2, INITIAL AND CONTINUING CALIBRATION VERIFICATION

When completed, WQP Form 2 contains analyte results and recoveries for all initial and continuing calibration verifications (ICVs, CCVs) performed for all requested parameters.

4.1. General Instructions for WQP Form 2

- 4.1.1. Units for values of each parameter reported in the *Found* and *True* columns shall be the same as the units reported on Form 1 for that parameter.
- 4.1.2. WQP Form 2 must be completed for all requested parameters except those determined by gravimetric methods and BOD/CBOD.

- 4.2. **Initial Calibration True:** In the *True* column under *Initial Calibration*, enter the certified value for each parameter analyzed. The number of significant figures reported shall be at least three. If the value certified by the supplier contains fewer than three significant figures, report results to the number of significant figures in the certified value.
- 4.3. **Initial Calibration Found:** In the *Found* column under *Initial Calibration*, enter the measured value for each parameter analyzed. Report this result to three significant figures.
- 4.4. **Initial Calibration %R:** Complete the %R column under *Initial Calibration* as follows:
- 4.4.1. For all required parameters except pH, enter the value of the ICV percent recovery to one decimal place. Compute %R according to the following equation:
- $$\%R = 100 * (\text{ICV Found}) \div (\text{ICV True})$$
- 4.4.2. For the parameter of pH, enter the Difference of the ICV Found and True values to two decimal places. Compute the Difference according to the following equation:
- $$\text{Difference} = (\text{ICV Found}) - (\text{ICV True})$$
- 4.5. **Continuing Calibration True:** In the *True* column under *Continuing Calibration*, enter the value for each parameter analyzed. The number of significant figures reported shall be at least three.
- 4.6. **Continuing Calibration Found:** In the *Found* columns under *Continuing Calibration*, enter the measured value for each parameter analyzed. Report this result to three significant figures.
- 4.7. **Continuing Calibration %R:** Complete the %R column under *Continuing Calibration* as follows:
- 4.7.1. For all required parameters except pH, enter the value of the CCV percent recovery to one decimal place. Compute %R according to the following equation:
- $$\%R = 100 * (\text{CCV Found}) \div (\text{CCV True})$$
- 4.7.2. For the parameter of pH, enter the Difference of the CCV Found and True values to two decimal places for pH. Compute the Difference according to the following equation:
- $$\text{Difference} = (\text{CCV Found}) - (\text{CCV True})$$
- 4.8. **Reporting Order:** The order of reporting ICVs and CCVs for each analyte shall follow the temporal order in which these standards were run with the first WQP Form 2 from left to right continuing to additional Forms 2 as appropriate.

5. WQP FORM 3, BLANKS

When completed, WQP Form 3 contains parameter results and qualifiers for initial calibration blanks, continuing calibration blanks, and preparation blanks (ICB, CCB, PB).

5.1. General Instructions for WQP Form 3

- 5.1.1. Units for all data reported in all *Result* columns shall be the same as the units reported on Form 1 for that parameter.

- 5.1.2. ICB and CCB data in WQP Form 3 must be completed for all requested parameters except conductivity, those parameters determined by gravimetric methods, pH, and BOD/CBOD (refer to SS06 Exhibit C Table C1 to determine method types for each approved method).
- 5.1.3. Preparation Blank data in WQP Form 3 must be completed when preparation is required for all requested parameters except conductivity, pH, alkalinity and acidity.
- 5.2. **MDL:** In the MDL column, enter the value of the MDL for each parameter as determined according to SS06 Exhibit D Section 11.
- 5.3. **Initial Calibration Blank:** To complete the column labeled *Initial Calibration Blank* enter measured values for each requested parameter as follows:
 - 5.3.1. If the absolute value of the ICB is less than the MDL, enter the MDL in the *Result* column and enter a *U* in the *C* column.
 - 5.3.2. If the absolute value of the ICB result is equal to or greater than the MDL and less than the RDL, enter actual results in the *Result* column and enter a *B* in the *C* column. Report results to three significant figures; negative values are entered with a minus (-) sign preceding the result.
 - 5.3.3. If the absolute value of the ICB result is greater than the RDL, enter actual results in the *Result* column and leave the *C* column blank. Report results to three significant figures; negative values are entered with a minus (-) sign preceding the result.
- 5.4. **Continuing Calibration Blank:** In the *Continuing Calibration Blank* columns, enter the measured value of each parameter in CCBs as follows:
 - 5.4.1. If the absolute value of the CCB is less than the MDL, enter the MDL in the *Result* column and enter a *U* in the *C* column.
 - 5.4.2. If the absolute value of the CCB result is equal to or greater than the MDL and less than the RDL, enter actual results in the *Result* column and enter a *B* in the *C* column. Report results to three significant figures; negative values are entered with a minus (-) sign preceding the result.
 - 5.4.3. If the absolute value of the CCB result is greater than the RDL, enter actual results in the *Result* column and leave the *C* column blank. Report results to three significant figures; negative values are entered with a minus (-) sign preceding the result.
- 5.5. **Reporting Order for ICBs and CCBs:** The order of reporting ICBs and CCBs for each parameter shall follow the temporal order in which these standards were run with the first WQP Form 3 from left to right continuing to additional Forms 3 as appropriate.
- 5.6. **Preparation Blank:** In the *Preparation Blank* column, enter the measured value of each parameter in the preparation blank as follows:
 - 5.6.1. If the absolute value of the PB result is less than the MDL, enter the MDL in the *Result* column and enter a *U* in the *C* column.
 - 5.6.2. If the absolute value of the PB result is equal to or greater than the MDL, enter actual results in the *Result* column and enter *B* in the *C* column. Report results to three significant figures; negative values are entered with a minus (-) sign preceding the result.

- 5.6.3. If the absolute value of the PB result is greater than the RDL, enter actual results in the *Result* column and leave the *C* column blank. Report results to three significant figures; negative values are entered with a minus (-) sign preceding the result.

6. WQP FORM 4, SPIKE SAMPLE RECOVERY

When completed, WQP Form 4 contains spike sample results, sample results, spike additions and recoveries for applicable parameters. Spike recovery qualifiers are assigned to data based on an evaluation of spike recoveries and sample analyte concentrations. These qualifiers are then entered in WQP Form 1.

6.1. General Instructions for WQP Form 4

- 6.1.1. Units for all data reported in all *Spiked Sample Result*, *Sample Result*, and *Spike Added* columns shall be the same as the units reported on Form 1 for that parameter.
- 6.1.2. WQP Form 4A must be completed for all requested parameters except pH, conductivity, BOD/CBOD and those parameters determined by gravimetric methods (refer to SS06 Exhibit C Table C1 to determine method types for approved methods of each parameter).
- 6.1.3. Use Form 4A-1 for both an analysis spike and preparation spike or sample-spike taken completely through both preparation and analysis steps. If more than one spike-sample analysis was completed for either a preparation or analysis batch, complete additional Form 4A's and identify Form 4A's as Form 4A-1, 4A-2,...etc.
- 6.1.4. Use Form 4B-1 for post preparation spikes or analysis spikes of prepared samples solutions. If more than one post preparation spike-sample analysis was completed for a preparation batch, complete additional Form 4B's and identify Form 4B's as Form 4B-1, 4B-2,...etc.
- 6.2. **Control Limit:** To complete the *Control Limit %R* column enter control limits for each parameter as follows:
- 6.2.1. If the results reported in the Sample Result column of Form 4 are less than or equal to four times the number reported in the Spike Added column, enter '75-125%' in the Control Limit %R column.
- 6.2.2. If the results reported in the Sample Result column of Form 4 are more than four times the number reported in the Spike Added column, leave the Control Limit column of WQP Form 4 empty.
- 6.3. **Spiked Sample Result (SSR):** In the *Spiked Sample Result* column of WQP Form 4, enter the measured value, corrected for dilutions, for the sample-spike analyzed according to SS06 Exhibit D Section 8.
- 6.4. **Sample Result (SR):** In the *Sample Result* and *C* columns of WQP Form 4, enter measured values, corrected for dilutions for the indicated sample as entered in the *Result* column of WQP Form 1. Note, the 'original sample' results are entered even if the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis.
- 6.5. **Spike Added (SA):** Enter the value for the analyte concentration added to the sample, corrected for dilutions.

- 6.6. **% Recovery:** Complete the column labeled %R as follows:
- 6.6.1. Enter the value to one decimal place of the percent recovery for all spiked analytes computed according to the following equation (see 6.6.2 & 6.6.3 below):
$$\%R = 100 * (SSR - SR) \div (SA)$$
 - 6.6.2. Spike calculations must be performed using the results of the sample designated as the 'original sample' (see Duplicate Sample Analysis). The average of sample and duplicate results must not be used for the purpose of determining percent recovery.
 - 6.6.3. When results are reported in the *Sample Result (SR)* column as less than the MDL, use SR = 0 only for purposes of calculating % Recovery.
 - 6.6.4. % Recovery shall be reported, without exception, for all parameters spiked
- 6.7. **Q Column:** Enter 'N' in the Q column, if the spike recovery (%R) is out of the control limits and the sample concentration is less than or equal to four times the spike amount. Also, if an 'N' is required in the Form 4 Q column, enter an 'N' for in the Form I Q column of all samples associated with the sample-spike matrix.

7. WQP FORM 5, DUPLICATE SAMPLE ANALYSIS

When completed, WQP Form 5 contains original and duplicate sample results, and a duplicate agreement qualifier, '*' when the difference between sample and duplicate sample results is not acceptable. If a qualifier is required, then the qualifier is also entered in WQP Form 1 Q column for all samples associated with the duplicate sample matrix.

7.1. General Instructions for WQP Form 5

- 7.1.1. Units for all data reported in all *Sample Result* and *Duplicate Result* columns shall be the same as the units reported on Form 1 for that parameter.
- 7.1.2. WQP Form 5 must be completed for all requested parameters.

7.2. Control Limit: Complete the column labeled *Control Limit* as follows:

- 7.2.1. For each requested parameter except pH and conductivity, enter the RDL if results reported in the *Sample Result* or *Duplicate Result* column of Form 5 are less than or equal to five times the RDL.
- 7.2.2. For each requested parameter except pH and conductivity, enter 20% in the *Control Limit* column of WQP Form 5, if the results reported in the *Sample Result* and *Duplicate Result* column of Form 5 are greater than or equal to five times the RDL.
- 7.2.3. For pH, enter 0.1 S.U.
- 7.2.4. For conductivity, enter 5%.

- 7.3. **Sample Result (SR):** Enter measured values, corrected for dilutions, (to a minimum of three significant figures) and concentration qualifiers for the indicated sample in the *Sample Result* and C columns of WQP Form 5 as entered in the *Result* column of WQP Form 1
- 7.4. **Duplicate Result (D):** Enter measured values, corrected for dilutions, (to a minimum of three significant figures) and concentration qualifiers for the duplicate sample analyzed according to SS06 Exhibit D Section 9 in the *Duplicate Result* and C columns of WQP Form 5 using the Form 1 guidelines for entering results and C column qualifiers on WQP Form 1.

7.5. **RPD:** For all parameters except pH and conductivity, complete the column labeled *RPD* as follows:

7.5.1. If either the sample or duplicate result is more than five times the parameter MDL, enter the absolute value of the Relative Percent Difference to one decimal place computed according to the following equation:

$$RPD = 100 * |SR - D| \div (SR + D)/2)$$

7.5.2. If both the sample and duplicate results are less than five times the analyte MDL, enter the absolute value of the Difference between the sample and duplicate sample results (see 7.5.3 below) to three decimal places computed according to the following equation:

$$\text{Difference} = |SR - D|$$

7.5.3. A value of zero shall be substituted for SR or D results which are reported as less than the MDL. However, if both SR and D are reported as less than the MDL, leave the RPD field empty.

7.6. **RPD:** For the parameter of pH and conductivity, complete the column labeled *RPD* as follows:

7.6.1. For pH, enter the absolute value of the Difference between the sample and duplicate sample results to two decimal places computed according to the following equation:

$$\text{Difference} = |SR - D|$$

7.6.2. For conductivity, enter the absolute value of the Relative Percent Difference to one decimal place computed according to the following equation:

$$RPD = 100 * |SR - D| \div (SR + D)/2)$$

7.7. **Q Column:** A “*” must be in the *Q* column to indicate duplicate results which are not within the control limits entered in the Form 5 *Control Limit* column. Enter “*” in the *Q* column if:

7.7.1. both sample and duplicate values are greater than or equal to 5 times the RDL and the RPD is greater than 20%, for all parameters except pH and conductivity.

7.7.2. either sample or duplicate values are less than 5 times the RDL and the absolute difference between the two values is greater than the RDL, for all parameters except pH and conductivity.

7.7.3. the sample and duplicate pH results have an absolute difference of more than 0.1 S.U.

7.7.4. the sample and duplicate conductivity results have an RPD greater than 5%.

8. **WQP FORM 6, LABORATORY CONTROL SAMPLE**

When completed WQP Form 6 contains results and recoveries for Laboratory Control Samples for all reported parameters.

8.1. **General Instructions for WQP Form 6**

8.1.1. Units for all data reported in all *Found* and *True* columns shall be the same as the units reported on Form 1 for that parameter.

8.1.2. Form 6 must be completed for all requested parameters.

- 8.2. **True:** Enter the certified true value for each parameter analyzed according to SS06 Exhibit D Section 8, in the *Found* column of WQP Form 6. If the true value is supplied as a range, enter the average of the range, or the supplied 'most probable' value. Report these values to the number significant figures to which they are reported on the certificate.
- 8.3. **Found:** Enter the measured values of each analyte found in the LCS according to SS06 Exhibit D Section 8 in the *Found* column of WQP Form 6. Report the result to a minimum of three significant figures.
- 8.4. **% Recovery:** Complete the column labeled %R as follows:
- 8.4.1. For all parameters except pH, enter the percent recovery to one decimal place for all analytes computed according to the following equation:
- $$\%R = 100 * (\text{Found}) \div (\text{True})$$
- 8.4.2. For pH, enter the absolute value of the Difference between the LCS True and Found values to two decimal places computed according to the following equation:
- $$\text{Difference} = |\text{Found} - \text{True}|$$
- 8.5. **Lower Control Limit %R:** Complete the *Lower Control Limit %R* column as follows:
- 8.5.1. For BOD and CBOD enter '82.0'.
- 8.5.2. For pH, enter '*True - 0.1 S.U.*'
- 8.5.3. For conductivity, enter '95.0'.
- 8.5.4. For all other requested parameters enter '80.0'.
- 8.6. **Upper Control Limit %R:** Complete the *Upper Control Limit %R* column as follows:
- 8.6.1. For BOD and CBOD enter '118.0'.
- 8.6.2. For pH, enter '*True + 0.1 S.U.*'
- 8.6.3. For conductivity, enter '105.0'.
- 8.6.4. For all other requested parameters enter '120.0'.
- 8.6.5. **BOD/CBOD Laboratory Control Sample Qualifier:** If %R for BOD/CBOD is outside of control limits, an 'E' qualifier must be entered on Form 1 for all samples in the analytical batch.

9. WQP HOLDING TIME SUMMARY

When completed, WQP Form 7 contains sample holding time information for each sample and associated analytical batch QC samples reported in the RIN to show whether sample holding times were met. Complete each of the Form 7 columns as follow:

- 9.1. **Date & Time Entries:** When actual sample hold time is within 24 hours of the maximum hold time, both date and time must be entered in a 24 hour format for the COC Sample Date, Preparation Date, and Analysis Date. And then determine and enter Preparation and Analysis Hold Times in days and hours. If a sample hold time is more than 24 hours less than the maximum hold time only the date need be entered.

- 9.2. **Site and Lab Sample Identifiers:** Enter at least one of the sample identifiers for each sample and each associated analytical batch QC sample in the appropriate Sample Identifier column.
- 9.3. **COC Sample Date/Time:** Enter the sampling date (dd/mmm/yy) and time (24 hour format) from the external COC.. Enter N/A for QC samples other than duplicates and spikes.
- 9.4. **VTSR Date:** Enter the VTSR date (dd/mmm/yy) from the external COC, for all samples. Enter N/A for QC samples other than duplicates and spikes.
- 9.5. **Preparation Date/Time:** Enter the date (dd/mmm/yy) and time (24 hr. format) that the sample preparation was started, if applicable. Enter N/A if no preparation was required.
- 9.6. **Preparation Holding Time:** Complete as follows:
- 9.6.1. If preparation was completed, enter the preparation holding time calculated as follows:
Preparation Hold Time (days/hours) = (Preparation Date/Time) - (COC Sample Date/Time)
- 9.6.2. Enter N/A if no preparation was required
- 9.7. **Analysis Date/Time:** Enter the date (dd/mmm/yy) and time (24 hr. format) that the sample analysis was started.
- 9.8. **Analysis Holding Time:** Complete as follows:
- 9.8.1. If preparation was completed, enter the analysis holding time calculated as follows:
Analysis Hold Time (days/hours) = (Analysis Date/Time) - (Preparation Date/Time)
- 9.8.2. If only analysis was completed without preparation, enter the analysis holding time calculated as follows:
Analysis Hold Time (days/hours) = (Analysis Date/Time) - (COC Sample Date/Time)
- 9.9. **Holding Times Met?:** Complete as follows:
- 9.9.1. Enter Yes or Y if both the Preparation Hold Time, if applicable and Analysis Hold Time are less than or equal to the Maximum Preparation and Analysis Hold Times.
- 9.9.2. Enter No or N if either the Preparation Hold Time, if applicable and Analysis Hold Time are greater than the Maximum Preparation and Analysis Hold Times.

WATER QUALITY PARAMETERS REPORTING FORM I
WQP ANALYSIS DATA SHEET

Lab Name: _____ RF Sample ID: _____

Lab Code: _____ Lab Sample ID: _____

RIN: _____

Parameter ID	Parameter Name	Result	Qualifiers		Units
			C ⁽¹⁾	Q	
10-70-8	Acidity				mg/L
T-005	Alkalinity, Total as CaCO ₃				mg/L
71-52-3	Alkalinity, Bicarbonate as CaCO ₃				mg/L
3812-32-6	Alkalinity, Carbonate as CaCO ₃				mg/L
7727-37-9	Ammonia as N				mg/L
10-26-4	BOD				mg/L
24959-67-9	Bromide				mg/L
11-03-0	CBOD				mg/L
C-004	COD				mg/L
16887-00-6	Chloride				mg/L
18540-29-9	Chromium VI				mg/L
57-12-5	Cyanide, Total				mg/L
10-87-7	Cyanide, Amenable to Chlorination				mg/L
10-71-9	Cyanide, Releasable, for RCRA Compliance				mg/L
16984-48-8	Fluoride				mg/L
11-02-9	Hardness as CaCO ₃				mg/L
14797-55-8	Nitrate as N				mg/L
14797-65-0	Nitrite as N				mg/L
C-005	Nitrate/Nitrite as N				mg/L
10-30-0	Oil and Grease, Total Recoverable				mg/L
11-59-6	Organic Carbon, Dissolved				mg/L
10-35-5	Organic Carbon, Total				mg/L
10-29-7	pH				S.U. at 25°C
108-95-2	Phenol				mg/L
14265-44-2	Phosphate (ortho) as P				mg/L
7723-14-0	Phosphate (total) as P				mg/L
RFS-SS-96	Sediment Analysis, Sand-Silt Split				mg/L
7631-86-9	Silica as SiO ₂ , Dissolved				mg/L
11-06-3	Solids, Non-Volatile Suspended (NVSS)				mg/L
C-008	Solids, Total (TS)				mg/L
10-33-3	Solids, Total Dissolved Solids (TDS)				mg/L
10-32-2	Solids, Total Suspended (TSS)				mg/L
10-34-4	Specific Conductance (Conductivity)				mmho/cm at 25°C
14808-79-8	Sulfate as SO ₄ ²⁻				mg/L
RFS-RS-97	Sulfide as H ₂ S				mg/L
18496-25-8	Sulfide as S				mg/L
7727-37-9	Total Kjeldahl Nitrogen (TKN)				mg/L
59473-04-0	Total Organic Halides (TOX)				mg/L
10-90-2	Total Petroleum Hydrocarbons (TPH)				mg/L
10-08-02	Turbidity				NTU

⁽¹⁾ A *U* in the *C* qualifier column indicates that this parameter was not detected; the method detection limit (MDL) has been entered in the *Result* column. A *B* in the *C* qualifier column indicates that this parameter was less than the RDL but greater than or equal to the MDL.

FORM I - WQP

WATER QUALITY PARAMETERS REPORTING FORM 2
INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: _____

RIN: _____

Lab Code: _____

Reporting units are as listed on Form I.

Parameter ID	Parameter Name	Initial Calibration (ICV)			Continuing Calibration (CCV)				
		True	Found	%R ⁽¹⁾	True	Found	%R ⁽¹⁾	Found	%R ⁽¹⁾
10-70-8	Acidity								
T-005	Alkalinity, Total as CaCO ₃								
71-52-3	Alkalinity, Bicarbonate as CaCO ₃								
3812-32-6	Alkalinity, Carbonate as CaCO ₃								
7727-37-9	Ammonia as N								
10-26-4	BOD	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
24959-67-9	Bromide								
11-03-0	CBOD	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
C-004	COD								
16887-00-6	Chloride								
18540-29-9	Chromium VI								
57-12-5	Cyanide, Total								
10-87-7	Cyanide, Amenable to Chlorination								
10-71-9	Cyanide, Releasable, for RCRA Compliance								
16984-48-8	Fluoride								
11-02-9	Hardness as CaCO ₃								
14797-55-8	Nitrate as N								
14797-65-0	Nitrite as N								
C-005	Nitrate/Nitrite as N								
10-30-0	Oil and Grease, Total Recoverable	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
11-59-6	Organic Carbon, Dissolved								
10-35-5	Organic Carbon, Total								
10-29-7	pH								
108-95-2	Phenol								
14265-44-2	Phosphate (ortho) as P								
7723-14-0	Phosphate (total) as P								
RFS-SS-96	Sediment Analysis, Sand-Silt Split	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
7631-86-9	Silica as SiO ₂ , Dissolved								
11-06-3	Solids, Non-Volatile Suspended (NVSS)	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
C-008	Solids, Total (TS)	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
10-33-3	Solids, Total Dissolved Solids (TDS)	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
10-32-2	Solids, Total Suspended (TSS)	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
10-34-4	Specific Conductance (Conductivity)								
14808-79-8	Sulfate as SO ₄ ²⁻								
RFS-RS-97	Sulfide as H ₂ S								
18496-25-8	Sulfide as S								
7727-37-9	Total Kjeldahl Nitrogen								
57473-04-0	Total Organic Halides (TOX)								
10-90-2	Total Petroleum Hydrocarbons (TPH)								
10-08-02	Turbidity								

N/A indicates information which is not applicable to this parameter.

⁽¹⁾ For pH enter |Found - True| in S.U.; for all other parameters enter %R = 100 * (Found) / (True)

FORM 2 - WQP

WATER QUALITY PARAMETERS REPORTING FORM 3
BLANKS

Lab Name: _____

RIN: _____

Lab Code: _____

Reporting units are as listed on Form I.

Parameter ID	Parameter Name	MDL	Initial Calibration Blank		Continuing Calibration Blank						Preparation Blank	
			Result	C	(1)		(2)		(3)		Result	C
10-70-8	Acidity										N/A	
T-005	Alkalinity, Total as CaCO ₃										N/A	
71-52-3	Alkalinity, Bicarbonate as CaCO ₃										N/A	
3812-32-6	Alkalinity, Carbonate as CaCO ₃										N/A	
7727-37-9	Ammonia as N											
10-26-4	BOD		N/A									
24959-67-9	Bromide											
11-03-0	CBOD		N/A									
C-004	COD											
16887-00-6	Chloride											
18540-29-9	Chromium VI											
57-12-5	Cyanide, Total											
10-87-7	Cyanide, Amenable to Chlorination											
10-71-9	Cyanide, Releasable, for RCRA Compliance											
16984-48-8	Fluoride											
11-02-9	Hardness as CaCO ₃											
14797-55-8	Nitrate as N											
14797-65-0	Nitrite as N											
C-005	Nitrate/Nitrite as N											
10-30-0	Oil and Grease, Total Recoverable		N/A									
11-59-6	Organic Carbon, Dissolved											
10-35-5	Organic Carbon, Total											
10-29-7	pH		N/A								N/A	
108-95-2	Phenol											
14265-44-2	Phosphate (ortho) as P											
7723-14-0	Phosphate (total) as P											
RFS-SS-96	Sediment Analysis, Sand-Silt Split		N/A									
7631-86-9	Silica as SiO ₂ , Dissolved											
11-06-3	Solids, Non-Volatile Suspended (NVSS)		N/A									
C-008	Solids, Total (TS)		N/A									
10-33-3	Solids, Total Dissolved Solids (TDS)		N/A									
10-32-2	Solids, Total Suspended (TSS)		N/A									
10-34-4	Specific Conductance (Conductivity)		N/A								N/A	
14808-79-8	Sulfate as SO ₄ ²⁻											
RFS-RS-97	Sulfide as H ₂ S											
18496-25-8	Sulfide as S											
7727-37-9	Total Kjeldahl Nitrogen											
59473-04-0	Total Organic Halides (TOX)											
10-90-2	Total Petroleum Hydrocarbons (TPH)											
10-08-02	Turbidity											

N/A indicates information which is not applicable to this parameter.

Control Limits: The absolute value of the blank must be less than the RDL

FORM 3 - WQP

WATER QUALITY PARAMETERS REPORTING FORM 4
SPIKE SAMPLE RECOVERY

Lab Name: _____

RIN: _____

Lab Code: _____

Site Sample Identifier: _____

Lab Sample Identifier: _____

Reporting units are as listed on Form I.

Parameter ID	Parameter Name	Control Limit ⁽¹⁾	Spiked Sample Result (SSR)	Sample Result (SR)	C	Spike Added (SA)	%R ⁽²⁾	Q
10-70-8	Acidity							
T-005	Alkalinity, Total as CaCO ₃							
71-52-3	Alkalinity, Bicarbonate as CaCO ₃							
3812-32-6	Alkalinity, Carbonate as CaCO ₃							
7727-37-9	Ammonia as N							
10-26-4	BOD	N/A	N/A	N/A		N/A	N/A	
24959-67-9	Bromide							
11-03-0	CBOD	N/A	N/A	N/A		N/A	N/A	
C-004	COD							
16887-00-6	Chloride							
18540-29-9	Chromium VI							
57-12-5	Cyanide, Total							
10-87-7	Cyanide, Amenable to Chlorination							
10-71-9	Cyanide, Releasable, for RCRA Compliance							
16984-48-8	Fluoride							
11-02-9	Hardness as CaCO ₃							
14797-55-8	Nitrate as N							
14797-65-0	Nitrite as N							
C-005	Nitrate/Nitrite as N							
10-30-0	Oil and Grease, Total Recoverable	N/A	N/A	N/A		N/A	N/A	
11-59-6	Organic Carbon, Dissolved							
10-35-5	Organic Carbon, Total							
10-29-7	pH	N/A	N/A	N/A		N/A	N/A	
108-95-2	Phenol							
14265-44-2	Phosphate (ortho) as P							
7723-14-0	Phosphate (total) as P							
RFS-SS-96	Sediment Analysis, Sand-Silt Split	N/A	N/A	N/A		N/A	N/A	
7631-86-9	Silica as SiO ₂ , Dissolved							
11-06-3	Solids, Non-Volatile Suspended (NVSS)	N/A	N/A	N/A		N/A	N/A	
C-008	Solids, Total (TS)	N/A	N/A	N/A		N/A	N/A	
10-33-3	Solids, Total Dissolved Solids (TDS)	N/A	N/A	N/A		N/A	N/A	
10-32-2	Solids, Total Suspended (TSS)	N/A	N/A	N/A		N/A	N/A	
10-34-4	Specific Conductance (Conductivity)	N/A	N/A	N/A		N/A	N/A	
14808-79-8	Sulfate as SO ₄ ²⁻							
RFS-RS-97	Sulfide as H ₂ S							
18496-25-8	Sulfide as S							
7727-37-9	Total Kjeldahl Nitrogen							
59473-04-0	Total Organic Halides (TOX)							
10-90-2	Total Petroleum Hydrocarbons (TPH)							
10-08-02	Turbidity							

N/A indicates information which is not applicable to this parameter.

⁽¹⁾ The *Control Limit* column is blank when SR > 4*SA and 75-125% when SR ≤ 4*SA.

⁽²⁾ %R = 100 * (SSR - SR) / (SA)

FORM 4 - WQP

WATER QUALITY PARAMETERS REPORTING FORM 5
DUPLICATE SAMPLE ANALYSIS

Lab Name: _____

RIN: _____

Lab Code: _____

Site Sample Identifier: _____

Lab Sample Identifier: _____

Reporting units are as listed on Form I.

Parameter ID	Parameter Name	Control Limit ⁽¹⁾	Sample Result (SR)	C	Duplicate Result (D)	C	RPD ⁽²⁾	Q
10-70-8	Acidity							
T-005	Alkalinity, Total as CaCO ₃							
71-52-3	Alkalinity, Bicarbonate as CaCO ₃							
3812-32-6	Alkalinity, Carbonate as CaCO ₃							
7727-37-9	Ammonia as N							
10-26-4	BOD							
24959-67-9	Bromide							
11-03-0	CBOD							
C-004	COD							
16887-00-6	Chloride							
18540-29-9	Chromium VI							
57-12-5	Cyanide, Total							
10-87-7	Cyanide, Amenable to Chlorination							
10-71-9	Cyanide, Releasable, for RCRA Compliance							
16984-48-8	Fluoride							
11-02-9	Hardness as CaCO ₃							
14797-55-8	Nitrate as N							
14797-65-0	Nitrite as N							
C-005	Nitrate/Nitrite as N							
10-30-0	Oil and Grease, Total Recoverable							
11-59-6	Organic Carbon, Dissolved							
10-35-5	Organic Carbon, Total							
10-29-7	pH	0.1 S.U.						
108-95-2	Phenol							
14265-44-2	Phosphate (ortho) as P							
7723-14-0	Phosphate (total) as P							
RFS-SS-96	Sediment Analysis, Sand-Silt Split							
7631-86-9	Silica as SiO ₂ , Dissolved							
11-06-3	Solids, Non-Volatile Suspended (NVSS)							
C-008	Solids, Total (TS)							
10-33-3	Solids, Total Dissolved Solids (TDS)							
10-32-2	Solids, Total Suspended (TSS)							
10-34-4	Specific Conductance (Conductivity)	5						
14808-79-8	Sulfate as SO ₄ ²⁻							
RFS-RS-97	Sulfide as H ₂ S							
18496-25-8	Sulfide as S							
7727-37-9	Total Kjeldahl Nitrogen							
59473-04-0	Total Organic Halides (TOX)							
10-90-2	Total Petroleum Hydrocarbons (TPH)							
10-08-02	Turbidity							

N/A indicates information which is not applicable to this parameter.

⁽¹⁾ The control limit for pH is 0.1 S.U.; conductivity is 5% RPD. For all other parameters enter 20% in the *control limit* column when SR > 5*MDL; enter the RDL when SR < 5*MDL.

⁽²⁾ RPD = 100 * |SR-D| / (SR+D)/2 units for RPD are percent

FORM 5 - WQP

WATER QUALITY PARAMETERS REPORTING FORM 6
LABORATORY CONTROL SAMPLE

Lab Name: _____

RIN: _____

Lab Code: _____

Reporting units are as listed on Form I.

Parameter ID	Parameter Name	True	Found	%R ⁽¹⁾	Lower Control Limit %R	Upper Control Limit %R
10-70-8	Acidity				80.0	120.0
T-005	Alkalinity, Total as CaCO ₃				80.0	120.0
71-52-3	Alkalinity, Bicarbonate as CaCO ₃				80.0	120.0
3812-32-6	Alkalinity, Carbonate as CaCO ₃				80.0	120.0
7727-37-9	Ammonia as N				80.0	120.0
10-26-4	BOD				82.0	118.0
24959-67-9	Bromide				80.0	120.0
11-03-0	CBOD				82.0	118.0
C-004	COD				80.0	120.0
16887-00-6	Chloride				80.0	120.0
18540-29-9	Chromium VI				80.0	120.0
57-12-5	Cyanide, Total				80.0	120.0
10-87-7	Cyanide, Amenable to Chlorination				80.0	120.0
10-71-9	Cyanide, Releasable, for RCRA Compliance				80.0	120.0
16984-48-8	Fluoride				80.0	120.0
11-02-9	Hardness as CaCO ₃				80.0	120.0
14797-55-8	Nitrate as N				80.0	120.0
14797-65-0	Nitrite as N				80.0	120.0
C-005	Nitrate/Nitrite as N				80.0	120.0
10-30-0	Oil and Grease, Total Recoverable				80.0	120.0
11-59-6	Organic Carbon, Dissolved				80.0	120.0
10-35-5	Organic Carbon, Total				80.0	120.0
10-29-7	pH				True-0.1 S.U.	True+0.1 S.U.
108-95-2	Phenol				80.0	120.0
14265-44-2	Phosphate (ortho) as P				80.0	120.0
7723-14-0	Phosphate (total) as P				80.0	120.0
RFS-SS-96	Sediment Analysis, Sand-Silt Split				80.0	120.0
7631-86-9	Silica as SiO ₂ , Dissolved				80.0	120.0
11-06-3	Solids, Non-Volatile Suspended (NVSS)				80.0	120.0
C-008	Solids, Total (TS)				80.0	120.0
10-33-3	Solids, Total Dissolved Solids (TDS)				80.0	120.0
10-32-2	Solids, Total Suspended (TSS)				80.0	120.0
10-34-4	Specific Conductance (Conductivity)				95.0	105.0
14808-79-8	Sulfate as SO ₄ ²⁻				80.0	120.0
RFS-RS-97	Sulfide as H ₂ S				80.0	120.0
18496-25-8	Sulfide as S				80.0	120.0
7727-37-9	Total Kjeldahl Nitrogen				80.0	120.0
59473-04-0	Total Organic Halides (TOX)				80.0	120.0
10-90-2	Total Petroleum Hydrocarbons (TPH)				80.0	120.0
10-08-02	Turbidity				80.0	120.0

N/A indicates information which is not applicable to this parameter.

⁽¹⁾ For pH enter |Found - True| in S.U.; for all other parameters enter %R = 100 * (Found) / (True)

⁽²⁾ Control Limits are stated as %R, except where units are noted.

FORM 6 - WQP

Lab Name: _____ RIN: _____
 Lab Code: _____ Parameter: _____
 Maximum Preparation Hold Time: _____
 Maximum Analysis Hold Time: _____

[illegible]

FORM 7 - WQP